Synthesis of Substituted Dihydrobenzofurans via Tandem S_NAr/5-*Exo*-*Trig*-Cyclization

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SUPPORTING INFORMATION

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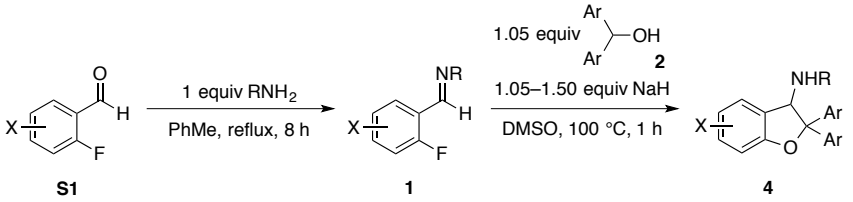
GENERAL INFORMATION

Unless otherwise noted, all materials were used as received from commercial sources without further purification. All aldehydes, amines, and diarylmethanols were purchased from Aldrich, Alfa Aesar, Acros, and Combi-Blocks. Bromodiphenylmethane, cesium carbonate, dry sodium hydride, and sodium *tert*-butoxide were purchased from Aldrich. All bulk solvents were purchased from VWR and used as received. Anhydrous DMSO was purchased from Aldrich. ^{1}H and ^{13}C spectra were recorded on a Varian Inova instrument (500 MHz and 125 MHz, respectively) or Bruker Ascend instrument with Prodigy broadband cryoprobe (400 MHz and 100 MHz, respectively), and ^{19}F NMR spectra were recorded on a Varian Mercury instrument (282 MHz). Spectra were internally referenced to $SiMe_4$ or solvent signals. The following abbreviations (or combinations thereof) were used to explain multiplicities: s = singlet, d = doublet, t = triplet, q = quartet, sept = septet, m = multiplet, and b = broad. High-resolution mass spectra (HRMS) were provided by the California Institute of Technology Mass Spectrometry Facility using a JEOL JMS-600H High Resolution Mass Spectrometer. All HRMS were ionized by EI or FAB.

EXPERIMENTAL PROCEDURES

General Two-Step S_NAr/5-exo-trig Cyclization Procedure:



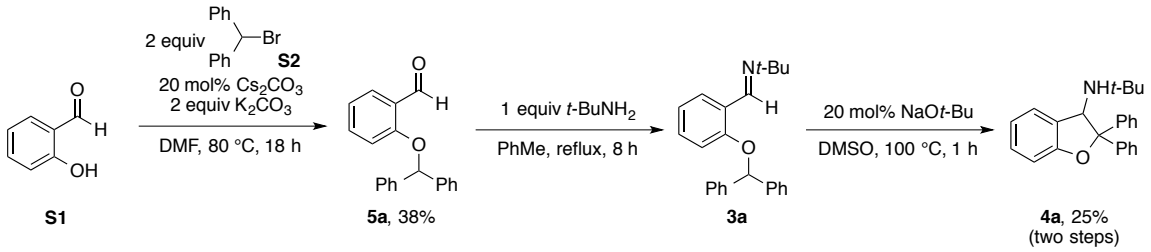
Scheme S1: General depiction of two-step S_NAr/5-*exo-trig* cyclization route for synthesizing 3-amino-2,3-dihydro-2,2-diarylbenzofurans.

General imine condensation procedure:[1] To a 100-mL round-bottom flask equipped with a Teflon-coated magnetic stir bar, were added the appropriate amine (10.0 mmol), the appropriate *ortho*-fluorobenzaldehyde (10.0 mmol), and toluene (50 mL). The flask was equipped with a Dean–Stark apparatus wrapped in aluminum foil and a reflux condenser. The reaction was allowed to stir at vigorous reflux (140–150 °C) for 8 h. During the course of the reaction, water accumulated at the bottom of the Dean–Stark apparatus. The reaction mixture was allowed to cool to room temperature. A small aliquot was taken, concentrated *in vacuo*, and analyzed by ^{1}H NMR to monitor reaction progress. (The *ortho*-fluorobenzaldehyde starting material possesses a characteristic ^{1}H NMR peak at 10.2–10.5 ppm (s) in $CDCl_3$, and the product possesses a characteristic ^{1}H NMR peak at 8.3–8.6 ppm (s); comparison of these two peaks provides a convenient means of monitoring reaction progress.) In cases where the reaction had not proceeded to >95% conversion, an additional portion of amine commensurate with the amount remaining starting material was added, and the reaction was heated for an additional 2–4 h. Upon completion, the reaction mixture was allowed to cool to room temperature, and the solvent was

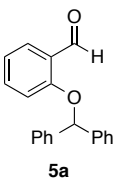
removed *in vacuo*. The crude imine product was obtained as a colorless/yellow oil or off-white solid and was used in the subsequent step without further purification.

General S_NAr/5-*exo-trig* cyclization procedure:[2] To a 100-mL Schlenk flask equipped with a Teflon-coated magnetic stir bar under Ar, were added dry NaH (252 mg, 10.5 mmol), DMSO (20 mL), and the appropriate alcohol (10.5 mmol). Upon addition of the alcohol, vigorous bubbling was observed. The solution was allowed to stir at room temperature for 1 h, during which time the reaction mixture became homogenous. A solution of the crude *ortho*-fluorobenzaldehyde imine from the previous step (assumed to be 10.0 mmol) in DMSO (10 mL) was added. The reaction mixture was heated to 100 °C for 1 h, during which time it changed color from yellow to red to brown. After 1 h, a small aliquot of the reaction mixture was removed with a syringe and quenched with H_2O. The resulting mixture was extracted with Et_2O, and the organic phase was concentrated *in vacuo* and examined by ^1H NMR spectroscopy to monitor reaction progress. (The starting material **1** has a characteristic ^1H NMR peak at 8.3–8.6 ppm (s, 1H) in $CDCl_3$, the intermediate **3** has a characteristic ^1H NMR peak at 8.7–8.9 ppm (s, 1H), and the product has a characteristic ^1H NMR peak at 4.8–5.9 (s or d, 1H); comparison of these three peaks provides a convenient means of monitoring reaction progress.) In instances where the reaction had not proceeded to completion (*i.e.*, >95% conversion) an additional portion of NaH was added, and the reaction mixture was heated at 100 °C for an additional 1 h. Upon completion of the reaction, the flask was allowed to cool to room temperature. The reaction mixture was carefully poured into a separatory funnel containing 100 mL of H_2O to quench residual base. The mixture was extracted with Et_2O (3 × 100 mL). The combined organic layers were washed with water (100 mL) and brine (100 mL), dried over Na_2SO_4, filtered, and concentrated *in vacuo*. The crude product was obtained as a yellow/brown oil or solid and was purified by silica gel column chromatography.

*Independent Synthesis of **3a** and Base-Mediated Cyclization:*

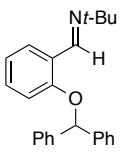


Scheme S2: Synthesis of **3a** and subsequent base-mediated cyclization.


5a

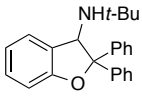
2-(benzhydryloxy)benzaldehyde (5a): To a 250-mL round-bottom flask equipped with a Teflon-coated magnetic stir bar under Ar were added salicylaldehyde (**S1**) (1.07 mL, 10.0 mmol), bromodiphenylmethane (**S2**) (4.94 g, 20.0 mmol), potassium carbonate (2.76 g, 20.0 mmol), cesium carbonate (0.65 g, 2.00 mmol) and DMF (100 mL). The reaction mixture was allowed to stir at 80 °C for 18 h. The flask was allowed to cool to room temperature, and the reaction was quenched with water and extracted with Et_2O (3 × 100 mL). The combined organic layers were washed with water (50 mL) and brine (50 mL), dried over Na_2SO_4, filtered, and concentrated *in vacuo*. The crude

product was obtained as a brown oil. Purification by silica gel column chromatography using 10:1 hexane:Et_2O as the eluent gave the product as an off-white solid (1.07 g, 37% yield). **^{1}H NMR** (500 MHz, $CDCl_3$) δ 10.66 (d, J = 0.8 Hz, 1H), 7.84 (dd, J_1 = 7.7 Hz, J_2 = 1.8 Hz, 1H), 7.46–7.42 (m, 4H), 7.41–7.34 (m, 4H), 7.32–7.27 (m, 1H), 6.33 (s, 1H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 189.97, 160.53, 140.59, 135.88, 128.96, 128.56, 128.25, 126.78, 125.73, 121.26, 114.87, 82.68; **HRMS** (EI+) *m/z* Calcd for $C_{20}H_{16}O_2$ $[M]^+$ 288.1150, found 288.1143.



3a

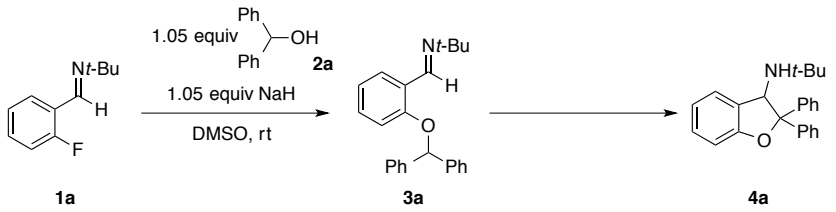
***N-tert*-butyl-1-(2-fluorophenyl)methanimine (3a):**[1] To a 25-mL round-bottom flask equipped with a Teflon-coated magnetic stir bar were added *tert*-butylamine (0.10 mL, 1.00 mmol), **5a** (288 mg, 1.00 mmol), and toluene (10 mL). The flask was equipped with a Dean–Stark apparatus wrapped in aluminum foil and a reflux condenser. The reaction was allowed to stir at vigorous reflux (140–150 °C) for 8 h. During the course of the reaction, water accumulated at the bottom of the Dean–Stark apparatus. The reaction was allowed to cool to room temperature. A small aliquot was taken, concentrated *in vacuo*, and analyzed by ^{1}H NMR to monitor reaction progress. (**5a** has a characteristic ^{1}H NMR peak at 10.66 ppm (d, J = 0.8 Hz, 1H) in $CDCl_3$, and the product (**3a**) has a ^{1}H NMR peak at 8.84 ppm (s, 1H); comparison of these two peaks provides a convenient means of monitoring reaction progress.) The reaction had proceeded to only 60% conversion, so an additional portion of *tert*-butylamine (0.16 mL, 1.50 mmol) was added, and the reaction was heated for a further 24 h. Upon completion, the reaction mixture was allowed to cool to room temperature, and the solvent was removed *in vacuo*. The crude imine product (**3a**) was obtained as a yellow oil and was used in the subsequent step without further purification. **^{1}H NMR** (300 MHz, $CDCl_3$) δ 8.84 (s, 1H), 7.93 (dd, J_1 = 7.7 Hz, J_2 = 1.8 Hz, 1H), 7.46–7.40 (m, 4H), 7.38–7.24 (m, 6H), 7.20 (ddd, J_1 = 8.2 Hz, J_2 = 7.5 Hz, J_3 = 1.8 Hz, 1H), 6.95 (t, J = 7.5 Hz, 1H), 6.85 (d, J = 8.6 Hz, 1H), 6.22 (s, 1H), 1.28 (s, 9H).



4a

***N*-(*tert*-butyl)-2,2-diphenyl-2,3-dihydrobenzofuran-3-amine (4a):** To a 10-mL Schlenk tube equipped with a Teflon-coated magnetic stir bar under Ar, were added dry NaO*t*-Bu (19 mg, 0.20 mmol) and a solution of **3a** (assumed to be 1.00 mmol) in DMSO (6 mL). The reaction mixture was heated to 100 °C for 1 h. The flask was allowed to cool to room temperature, and the reaction was carefully quenched with H_2O and extracted with Et_2O (3 × 50 mL). The organic layers were combined, washed with water (50 mL) and brine (50 mL), dried over Na_2SO_4, filtered, and concentrated *in vacuo*. Purification by silica gel column chromatography using 200:1 hexane:Et_2O as the eluent gave the product as a viscous yellow oil (87 mg, 25% yield). Analytical data were consistent with that of the sample of **4a** prepared by the general two-step S_NAr/5-*exo*-*trig* cyclization procedure.

S_NAr/5-exo-trig Cyclization Reaction Kinetics:



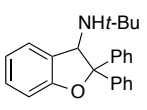
Scheme S3: S_NAr/5-*exo-trig* cyclization reaction monitored by ^1H NMR spectroscopy over time.

General Procedure for ^1H NMR Kinetics of Tandem S_NAr/5-*exo-trig* Cyclization: *N-tert*-Butyl-1-(2-fluorophenyl)methanimine (**1a**) (10.0 mmol) was prepared according to the general imine condensation procedure. To a 100-mL Schlenk flask equipped with a Teflon-coated magnetic stir bar under Ar, were added dry NaH (252 mg, 10.5 mmol), DMSO (20 mL), and diphenylmethanol (1.93 g, 10.5 mmol). Upon addition of the alcohol, vigorous bubbling was observed. The solution was allowed to stir at room temperature for 1 h, during which time the reaction mixture became homogenous. A solution of crude **1a** in DMSO (10 mL) was added, and the reaction mixture was allowed to stir at room temperature for 36 h, during which time a color change to yellow then red/brown was observed. At predetermined intervals, a small aliquot (approximately 0.5 mL) was removed and quenched with H_2O. The resulting mixture was extracted with Et_2O, the solvent was removed *in vacuo*, and a ^1H NMR spectrum was collected. Reaction progress was monitored by comparing the integrals of diagnostic peaks for **1a**, **3a**, and **4a** at 8.57 (s, 1H), 8.84 (s, 1H), and 4.90 (s, 1H), respectively. The results are shown in Figure 1 and Table S1.

Table S1: Reaction progress over time for the transformation depicted in Scheme S3.

	% (^1H NMR)		
time	**1a**	**3a**	**4a**
10 min	90	10	0
1 h	78	15	7
2 h	62	19	20
3 h	51	19	30
4 h	41	19	40
12 h	16	14	70
24 h	6	8	86
36 h	6	8	86

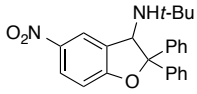
Characterization of New Compounds:



4a

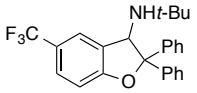
3-(*tert*-butyl)-2,2-diphenyl-3,4-dihydro-2*H*-benzo[*e*][1,3]oxazine (4a): The title compound was prepared from 2-fluorobenzaldehyde (1.05 mL, 10.0 mmol), *tert*-butylamine (1.04 mL, 10.0 mmol), and diphenylmethanol (1.93 g, 10.5 mmol) according to the general procedure. Purification by silica gel column

chromatography using 20:1 hexane:Et_2O as the eluent gave the product as a viscous yellow oil (1.37 g, 40% yield). **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.70–7.63 (m, 2H), 7.43–7.38 (m, 2H), 7.32 (t, J = 7.6 Hz, 2H), 7.28–7.19 (m, 5H), 7.14 (t, J = 7.7 Hz, 1H), 6.94 (d, J = 8.1 Hz, 1H), 6.88–6.83 (m, 1H), 4.90 (s, 1H), 0.92 (s, 9H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 158.43, 143.81, 140.31, 132.51, 129.16, 129.05, 128.17, 127.68, 127.41, 127.37, 127.01, 125.33, 121.34, 110.62, 95.96, 63.54, 51.04, 30.47; **HRMS** (FAB+) *m/z* Calcd for $C_{24}H_{25}ON$ $[M]^+$ 343.1936, found 343.1942.



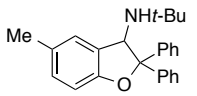
4b

***N*-(*tert*-butyl)-5-nitro-2,2-diphenyl-2,3-dihydrobenzofuran-3-amine (4b):** The title compound was prepared from 2-fluoro-5-nitrobenzaldehyde (1.69 g, 10.0 mmol), *tert*-butylamine (1.04 mL, 10.0 mmol), and diphenylmethanol (1.93 g, 10.5 mmol) according to the general procedure. An additional portion of NaH (48 mg, 2.0 mmol) was added because the cyclization had not proceeded to completion. After the first chromatographic purification on silica gel (20:1 hexane:Et_2O), the product still contained diphenylmethanol. A second purification by silica gel column chromatography (10:1 hexane:DCM → 1:1 hexane:DCM) provided the product as a white solid (660 mg, 17% yield). **^{1}H NMR** (500 MHz, $CDCl_3$) δ 8.23–8.12 (m, 2H), 7.42–7.21 (m, 11H), 6.09 (s, 1H), 1.84 (s, 1H), 1.28 (s, 9H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 152.03, 148.38, 145.26, 143.87, 143.58, 128.37, 128.28, 127.78, 127.77, 127.38, 127.37, 124.57, 124.06, 119.51, 89.97, 89.33, 51.12, 31.10; **HRMS** (FAB+) *m/z* Calcd for $C_{24}H_{25}N_2O_3$ $[M+H]^+$ 389.1865, found 389.1881.



4c

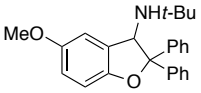
***N*-(*tert*-butyl)-2,2-diphenyl-5-(trifluoromethyl)-2,3-dihydrobenzofuran-3-amine (4c):** The title compound was prepared from 2-fluoro-5-(trifluoromethyl)benzaldehyde (1.41 mL, 10.0 mmol), *tert*-butylamine (1.04 mL, 10.0 mmol), and diphenylmethanol (1.93 g, 10.5 mmol) according to the general procedure. Purification by silica gel column chromatography using a gradient solvent system (40:1 hexane:Et_2O → 20:1 hexane:Et_2O) as the eluent gave the product as an off-white solid (3.13 g, 76% yield). **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.70–7.62 (m, 2H), 7.48 (s, 1H), 7.43 (dd, J_1 = 8.6 Hz, J_2 = 1.9 Hz, 1H), 7.40–7.32 (m, 4H), 7.32–7.22 (m, 4H), 6.99 (d, J = 8.4 Hz, 1H), 4.98 (s, 1H), 0.95 (s, 9H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 161.04, 143.17, 139.70, 133.51, 128.97, 128.38, 128.10, 127.77, 127.37, 127.32, 126.98 (q, J_{C-F} = 3.8 Hz), 124.69 (q, J_{C-F} = 270.0 Hz), 123.88 (q, J_{C-F} = 32.5 Hz), 122.97 (q, J_{C-F} = 3.8 Hz), 110.71, 97.34, 63.07, 51.24, 30.57; **^{19}F NMR** (282 MHz, $CDCl_3$) δ –60.96 (s, 3F); **HRMS** (FAB+) *m/z* Calcd for $C_{24}H_{24}F_3NO$ $[M]^+$ 411.1810, found 411.1817.



4d

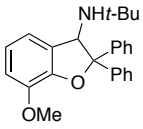
***N*-(*tert*-butyl)-5-methyl-2,2-diphenyl-2,3-dihydrobenzofuran-3-amine (4d):** The title compound was prepared from 2-fluoro-5-methylbenzaldehyde (1.22 mL, 10.0 mmol), *tert*-butylamine (1.04 mL, 10.0 mmol), and diphenylmethanol (1.93 g, 10.5 mmol) according to the general procedure. An additional portion of NaH (96 mg, 4.0 mmol) was added because the cyclization had not proceeded to completion. Purification by silica gel column chromatography using 40:1 hexane:Et_2O as the eluent gave the product as a colorless oil (2.42 g, 68% yield). **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.69–7.64 (m, 2H), 7.42–7.38 (m, 2H), 7.31 (t, J = 7.9 Hz, 2H), 7.27–7.17 (m,

4H), 7.01 (s, 1H), 6.93 (d, J = 8.1 Hz, 1H), 6.83 (d, J = 8.1 Hz, 1H), 4.85 (s, 1H), 2.23 (s, 3H), 0.91 (s, 9H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 156.36, 143.89, 140.38, 132.42, 130.52, 129.52, 129.20, 128.13, 127.62, 127.40, 127.32, 126.97, 125.73, 110.14, 95.95, 63.66, 51.00, 30.44, 21.06; **HRMS** (FAB+) *m/z* Calcd for $C_{25}H_{27}ON$ $[M]^+$ 357.2093, found 357.2094.



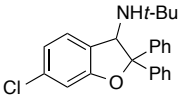
4e

***N*-(*tert*-butyl)-5-methoxy-2,2-diphenyl-2,3-dihydrobenzofuran-3-amine (4e):** The title compound was prepared from 2-fluoro-5-methoxybenzaldehyde (1.00 mL, 10.0 mmol), *tert*-butylamine (1.04 mL, 10.0 mmol), and diphenylmethanol (1.93 g, 10.5 mmol) according to the general procedure. An additional portion of NaH (192 mg, 8.0 mmol) was added because the cyclization had not proceeded to completion. Purification by silica gel column chromatography using a gradient solvent system (66:33:1 hexane:DCM:methanol → 80:20:1 hexane:DCM:methanol) as the eluent gave the product as a brown oil (821 mg, 22% yield). **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.68–7.64 (m, 2H), 7.43–7.39 (m, 2H), 7.36–7.32 (m, 2H), 7.29–7.21 (m, 6H), 6.85 (d, J = 8.6 Hz, 1H), 6.80 (d, J = 2.7 Hz, 1H), 6.70 (dd, J_1 = 8.6 Hz, J_2 =2.7 Hz, 1H), 4.85 (s, 1H), 3.73 (s, 3H), 0.93 (s, 9H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 154.65, 152.52, 143.91, 140.33, 133.49, 129.13, 128.19, 127.70, 127.42, 127.41, 127.09, 114.03, 111.45, 110.64, 96.17, 63.92, 56.10, 51.06, 30.47; **HRMS** (FAB+) *m/z* Calcd for $C_{25}H_{27}O_2N$ $[M]^+$ 373.2042, found 373.2036.



4f

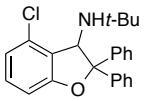
***N*-(*tert*-butyl)-7-methoxy-2,2-diphenyl-2,3-dihydrobenzofuran-3-amine (4f):** The title compound was prepared from 2-fluoro-3-methoxybenzaldehyde (1.54 g, 10.0 mmol), *tert*-butylamine (1.04 mL, 10.0 mmol), and diphenylmethanol (1.93 g, 10.5 mmol) according to the general procedure. An additional portion of NaH (72 mg, 3.0 mmol) was added because the cyclization had not proceeded to completion. Purification by silica gel column chromatography using a gradient solvent system (40:1 hexane:Et_2O → 10:1 hexane:Et_2O) as the eluent gave the product as a viscous pale-yellow oil (2.54 g, 68% yield). **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.72–7.66 (m, 2H), 7.44–7.38 (m, 2H), 7.36–7.29 (m, 2H), 7.28–7.18 (m, 4H), 6.86 (dd, J_1 = 7.3 Hz, J_2 = 0.8 Hz, 1H), 6.82 (t, J = 7.7 Hz, 1H), 6.75 (dd, J_1 = 7.9 Hz, J_2 = 1.3 Hz, 1H), 4.90 (s, 1H), 3.90 (s, 3H), 0.91 (s, 9H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 146.95, 145.15, 143.72, 139.99, 133.70, 129.30, 128.14, 127.69, 127.41, 127.34, 126.94, 121.85, 117.31, 112.18, 96.59, 63.78, 56.34, 51.01, 30.40; **HRMS** (FAB+) *m/z* Calcd for $C_{25}H_{27}NO_2$ $[M]^+$ 373.2042, found 373.2059.



4g

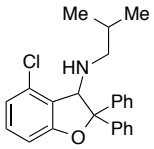
***N*-(*tert*-butyl)-6-chloro-2,2-diphenyl-2,3-dihydrobenzofuran-3-amine (4g):** The title compound was prepared from 4-chloro-2-fluorobenzaldehyde (1.59 g, 10.0 mmol), *tert*-butylamine (1.04 mL, 10.0 mmol), and diphenylmethanol (1.93 g, 10.5 mmol) according to the general procedure. An additional portion of NaH (120 mg, 5.0 mmol) was added because the cyclization had not proceeded to completion. After the first chromatographic purification on silica gel (6:1 hexane:DCM → 4:1 hexane:DCM), the product still contained unidentifiable impurities. A second purification by silica gel column chromatography (20:1 hexane:acetone) provided the product as a yellow solid (1.19 g, 31% yield). **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.67–7.57 (m, 2H), 7.39–7.31 (m, 4H), 7.30–7.20 (m, 4H), 7.13 (d, J = 8.0 Hz, 1H), 6.94 (d, J = 1.9 Hz, 1H), 6.84 (dd, J_1 = 8.0 Hz, J_2 = 1.9

Hz, 1H), 4.87 (s, 1H), 0.91 (s, 9H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 159.20, 143.28, 139.81, 134.32, 131.40, 128.96, 128.28, 127.93, 127.61, 127.30, 127.18, 126.02, 121.59, 111.34, 97.14, 62.83, 51.12, 30.46; **HRMS** (FAB+) *m/z* Calcd for $C_{24}H_{24}ClNO$ $[M]^+$ 377.1546, found 377.1549.



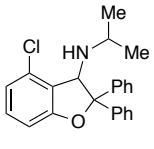
4h

***N*-(*tert*-butyl)-4-chloro-2,2-diphenyl-2,3-dihydrobenzofuran-3-amine (4h):** The title compound was prepared from 2-chloro-6-fluorobenzaldehyde (1.59 g, 10.0 mmol), *tert*-butylamine (1.04 mL, 10.0 mmol), and diphenylmethanol (1.93 g, 10.5 mmol) according to the general procedure. Purification by silica gel column chromatography using a gradient solvent system (40:1 hexane:Et_2O → 20:1 hexane:Et_2O) as the eluent gave the product as an off-white solid (3.35 g, 89% yield). **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.66–7.61 (m, 2H), 7.39–7.32 (m, 4H), 7.32–7.24 (m, 4H), 7.08 (t, J = 8.0 Hz, 1H), 6.85 (d, J = 8.0 Hz, 1H), 6.81 (dd, J_1 = 8.0 Hz, J_2 = 0.9 Hz, 1H), 4.92 (s, 1H), 0.78 (s, 9H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 159.80, 142.77, 140.14, 130.62, 130.29, 130.29, 130.15, 128.32, 127.92, 127.52, 127.00, 122.16, 109.27, 96.45, 63.50, 51.09, 30.32; **HRMS** (FAB+) *m/z* Calcd for $C_{24}H_{24}ClNO$ $[M]^+$ 377.1546, found 377.1549; **X-ray** (single-crystal) Crystals suitable for X-ray diffraction were grown by vapor diffusion of hexane into a saturated solution of the title compound in Et_2O at room temperature (CCDC 1058167).[3]



4i

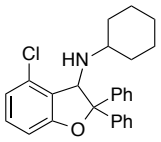
4-chloro-*N*-isobutyl-2,2-diphenyl-2,3-dihydrobenzofuran-3-amine (4i): The title compound was prepared from 2-chloro-6-fluorobenzaldehyde (1.59 g, 10.0 mmol), isobutylamine (0.73 g, 10.0 mmol), and diphenylmethanol (1.93 g, 10.5 mmol) according to the general procedure. An additional portion of NaH (48 mg, 2.0 mmol) was added because the cyclization had not proceeded to completion. Purification by silica gel column chromatography using 200:1 hexane:Et_2O as the eluent gave the product as a yellow oil (2.60 g, 69% yield). **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.60 (d, J = 7.7 Hz, 2H), 7.52 (d, J = 7.6 Hz, 2H), 7.31 (q, J = 6.8 Hz, 4H), 7.24 (q, J = 7.4 Hz, 2H), 7.13 (t, J = 8.0 Hz, 1H), 6.87 (t, J = 7.2 Hz, 2H), 4.97 (s, 1H), 2.13 (dd, J_1 = 10.7 Hz, J_2 = 6.3 Hz, 1H), 1.82 (dd, J_1 = 10.3 Hz, J_2 = 6.8 Hz, 1H), 1.40 (s, 1H), 1.34–1.23 (m, 1H), 0.58 (d, J = 6.6 Hz, 3H), 0.55 (d, J = 6.6 Hz, 3H); **^{13}C NMR** (100 MHz, $(CD_3)_2CO$) δ 160.49, 145.40, 141.45, 132.22, 131.72, 129.23, 128.83, 128.54, 128.49, 128.13, 128.06, 127.31, 122.38, 109.97, 96.36, 68.44, 54.51, 29.62, 20.76, 20.73; **HRMS** (FAB+) *m/z* Calcd for $C_{24}H_{24}ClNO$ $[M]^+$ 377.1546, found 377.1540.



4j

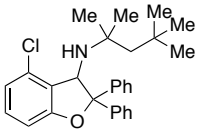
4-chloro-*N*-isopropyl-2,2-diphenyl-2,3-dihydrobenzofuran-3-amine (4j): The title compound was prepared from 2-chloro-6-fluorobenzaldehyde (1.59 g, 10.0 mmol), isopropylamine (0.85 mL, 10.0 mmol), and diphenylmethanol (1.93 g, 10.5 mmol) according to the general procedure. Purification by silica gel column chromatography using a gradient solvent system (200:1 hexane:Et_2O → 40:1 hexane:Et_2O) as the eluent gave the product as an off-white solid (2.57 g, 71% yield). **^{1}H NMR** (500 MHz, CD_2Cl_2) δ 7.86–7.81 (m, 2H), 7.67–7.63 (m, 2H), 7.55–7.38 (m, 6H), 7.25 (t, J = 8.0 Hz, 1H), 7.07–6.99 (m, 2H), 5.16 (s, 1H), 2.55 (sept, J = 6.2 Hz, 1H), 1.42 (s, 1H), 1.18 (d, J = 6.1 Hz, 3H), 0.67 (d, J = 6.3 Hz, 3H); **^{13}C NMR** (125 MHz, CD_2Cl_2) δ 159.87, 143.80, 140.46, 131.94, 131.10, 129.37, 128.94, 128.52, 128.47, 128.27, 128.22, 127.62,

122.34, 109.64, 96.61, 65.93, 47.44, 23.74, 22.93; **HRMS** (FAB+) *m/z* Calcd for $C_{23}H_{22}ClNO$ $[M]^+$ 363.1390, found 363.1405.



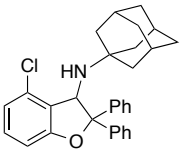
4k

4-chloro-*N*-cyclohexyl-2,2-diphenyl-2,3-dihydrobenzofuran-3-amine (4k): The title compound was prepared from 2-chloro-6-fluorobenzaldehyde (1.59 g, 10.0 mmol), cyclohexylamine (1.12 mL, 10.0 mmol), and diphenylmethanol (1.93 g, 10.5 mmol) according to the general procedure. Purification by silica gel column chromatography using 40:1 hexane:Et_2O as the eluent gave the product as an off-white solid (2.59 g, 64% yield). **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.63–7.59 (m, 2H), 7.48–7.42 (m, 2H), 7.36–7.32 (m, 2H), 7.31–7.23 (m, 4H), 7.07 (t, J = 8.0 Hz, 1H), 6.87–6.79 (m, 2H), 4.99 (s, 1H), 2.03–1.90 (m, 2H), 1.72–1.60 (m, 1H), 1.49–1.37 (m, 2H), 1.21 (bs, 1H), 1.16–1.05 (m, 1H), 1.06–0.94 (m, 2H), 0.90–0.79 (m, 1H), 0.79–0.72 (m, 1H), 0.70–0.60 (m, 1H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 159.28, 143.17, 139.87, 131.33, 130.47, 128.63, 128.36, 127.95, 127.91, 127.75, 127.67, 127.18, 121.90, 109.08, 96.12, 64.95, 55.11, 33.75, 33.22, 26.11, 25.22, 24.98; **HRMS** (FAB+) *m/z* Calcd for $C_{26}H_{26}ClNO$ $[M]^+$ 403.1703, found 403.1714.



4l

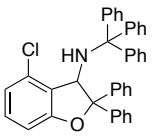
4-chloro-2,2-diphenyl-*N*-(2,4,4-trimethylpentan-2-yl)-2,3-dihydrobenzofuran-3-amine (4l): The title compound was prepared from 2-chloro-6-fluorobenzaldehyde (1.59 g, 10.0 mmol), *tert*-octylamine (1.60 mL, 10.0 mmol), and diphenylmethanol (1.93 g, 10.5 mmol) according to the general procedure. Purification by silica gel column chromatography using a gradient solvent system (200:1 hexane:Et_2O → 40:1 hexane:Et_2O) as the eluent gave the product as an off-white solid (2.77 g, 64% yield). **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.65–7.59 (m, 2H), 7.39–7.32 (m, 4H), 7.30–7.26 (m, 4H), 7.08 (t, J = 8.0 Hz, 1H), 6.84 (d, J = 8.0 Hz, 1H), 6.81 (dd, J_1 = 8.0 Hz, J_2 = 0.7 Hz, 1H), 4.97 (s, 1H), 1.22–1.07 (m, 5H), 0.85 (s, 9H), 0.53 (s, 3H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 159.69, 142.76, 140.28, 130.39, 130.37, 130.21, 130.09, 128.31, 127.86, 127.83, 127.42, 127.06, 122.16, 109.28, 96.52, 63.03, 56.85, 55.16, 31.92, 31.60, 29.10, 29.06; **HRMS** (FAB+) *m/z* Calcd for $C_{28}H_{32}ClNO$ $[M]^+$ 433.2172, found 433.2190.



4m

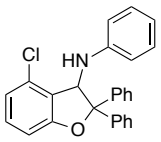
***N*-((1s,3s)-adamantan-1-yl)-4-chloro-2,2-diphenyl-2,3-dihydrobenzofuran-3-amine (4m):** The title compound was prepared from 2-chloro-6-fluorobenzaldehyde (1.59 g, 10.0 mmol), 1-adamantylamine (1.51 g, 10.0 mmol), and diphenylmethanol (1.93 g, 10.5 mmol) according to the general procedure. An additional portion of NaH (72 mg, 3.0 mmol) was added because the cyclization had not proceeded to completion. Purification by silica gel column chromatography using a gradient solvent system (200:1 hexane:Et_2O → 40:1 hexane:Et_2O) as the eluent gave the product as a white solid (3.54 g, 78% yield). **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.65–7.62 (m, 2H), 7.39–7.32 (m, 4H), 7.31–7.23 (m, 4H), 7.07 (t, J = 8.0 Hz, 1H), 6.84 (d, J = 7.9 Hz, 1H), 6.81 (dd, J_1 = 8.0 Hz, J_2 = 0.8 Hz, 1H), 5.04 (s, 1H), 1.88 (s, 3H), 1.52 (d, J = 12.3 Hz, 3H), 1.42 (d, J = 11.3 Hz, 3H), 1.37–1.29 (m, 3H), 1.15 (dq, J_1 = 11.9 Hz, J_2 = 2.8 Hz, 3H), 0.95 (s, 1H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 159.75, 142.71, 140.05, 130.52, 130.39, 130.22, 130.10, 128.29, 127.89, 127.88, 127.48, 126.98, 122.12, 109.23, 96.45, 61.60,

51.06, 43.80, 36.56, 29.81; **HRMS** (FAB+) *m/z* Calcd for $C_{30}H_{30}ClNO$ $[M]^+$ 455.2016, found 455.2001.



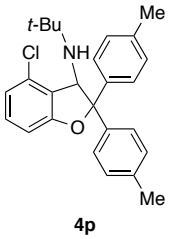
4n

4-chloro-2,2-diphenyl-*N*-trityl-2,3-dihydrobenzofuran-3-amine (4n): The title compound was prepared from 2-chloro-6-fluorobenzaldehyde (1.59 g, 10.0 mmol), triphenylmethylamine (2.59 g, 10.0 mmol), and diphenylmethanol (1.93 g, 10.5 mmol) according to the general procedure. An additional portion of NaH (72 mg, 3.0 mmol) was added because the cyclization had not proceeded to completion. Purification by silica gel column chromatography using a gradient solvent system (100:1 hexane:Et_2O → 40:1 hexane:Et_2O) as the eluent gave the product as a white solid (1.58 g, 28% yield). **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.35–6.98 (m, 28H), 6.87 (d, J = 7.9 Hz, 1H), 6.57 (dd, J_1 = 8.0 Hz, J_2 = 0.8 Hz, 1H), 4.92 (d, J = 6.5 Hz, 1H), 2.35 (d, J = 6.5 Hz, 1H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 159.31, 146.11, 143.26, 140.33, 131.99, 131.89, 130.04, 129.54, 128.20, 128.19, 127.59, 127.55, 127.52, 127.38, 126.60, 126.30, 121.52, 109.53, 98.52, 71.40, 59.99; **HRMS** (FAB+) *m/z* Calcd for $C_{39}H_{30}ClNO$ $[M]^+$ 563.2016, found 563.2032; **X-ray** (single-crystal) Colorless block crystals suitable for X-ray diffraction were grown from a solution of the title compound in 40:1 hexane:Et_2O at room temperature (CCDC 1058169).[3]



4o

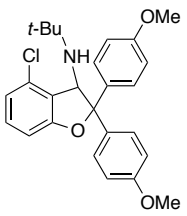
4-chloro-*N*,2,2-triphenyl-2,3-dihydrobenzofuran-3-amine (4o): The title compound was prepared from 2-chloro-6-fluorobenzaldehyde (1.59 g, 10.0 mmol), aniline (0.91 mL, 10.0 mmol), and diphenylmethanol (1.93 g, 10.5 mmol) according to the general procedure. An additional portion of NaH (96 mg, 4.0 mmol) was added because the cyclization had not proceeded to completion. Purification by silica gel column chromatography using 40:1 hexane:Et_2O as the eluent gave the product as a yellow oil (889 mg, 22% yield). **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.68–7.58 (m, 2H), 7.39–7.26 (m, 5H), 7.21–7.15 (m, 4H), 7.11–7.04 (m, 2H), 6.93 (d, J = 8.0 Hz, 1H), 6.90 (dd, J_1 = 8.0 Hz, J_2 = 0.7 Hz, 1H), 6.67 (tt, J_1 = 7.4 Hz, J_2 = 1.0 Hz, 1H), 6.51–6.47 (m, 2H), 5.86 (d, J = 10.4 Hz, 1H), 3.54 (d, J = 10.4 Hz, 1H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 159.51, 146.75, 143.19, 139.37, 132.08, 131.13, 129.19, 128.63, 128.17, 127.88, 127.82, 127.29, 126.76, 126.73, 122.17, 118.19, 113.73, 109.13, 96.08, 64.48; **HRMS** (FAB+) *m/z* Calcd for $C_{26}H_{20}ClNO$ $[M]^+$ 397.1233, found 397.1237.



4p

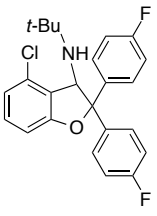
***N*-(*tert*-butyl)-4-chloro-2,2-di-*p*-tolyl-2,3-dihydrobenzofuran-3-amine (4p):** The title compound was prepared from 2-chloro-6-fluorobenzaldehyde (1.59 g, 10.0 mmol), *tert*-butylamine (1.04 mL, 10.0 mmol), and 4,4'-dimethylbenzhydrol (2.23 g, 10.5 mmol) according to the general procedure. An additional portion of NaH (120 mg, 5.0 mmol) was added because the cyclization had not proceeded to completion. Purification by silica gel column chromatography using a gradient solvent system (100:1 hexane:Et_2O → 40:1 hexane:Et_2O) as the eluent gave the product as an off-white solid (2.69 g, 66% yield). **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.53–7.48 (m, 2H), 7.25–7.20 (m, 2H), 7.17 (d, J = 8.0 Hz, 2H), 7.10–7.05 (m, 3H), 6.83 (d, J = 7.9 Hz, 1H), 6.81 (dd, J_1 = 8.0 Hz, J_2 = 0.8 Hz, 1H), 4.89 (s, 1H), 2.33 (s, 6H), 0.80 (s, 9H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 159.77, 139.87, 137.46, 137.26, 130.56, 130.36, 130.18, 130.00, 128.99, 128.98,

127.70, 127.34, 122.00, 109.19, 96.32, 63.22, 51.09, 30.31, 21.24, 21.19; **HRMS** (FAB+) *m/z* Calcd for $C_{26}H_{28}ClNO$ $[M]^+$ 405.1859, found 405.1986.



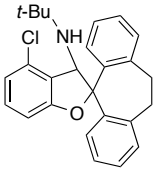
4q

***N*-(*tert*-butyl)-4-chloro-2,2-bis(4-methoxyphenyl)-2,3-dihydrobenzofuran-3-amine (4q):** The title compound was prepared from 2-chloro-6-fluorobenzaldehyde (1.59 g, 10.0 mmol), *tert*-butylamine (1.04 mL, 10.0 mmol), and 4,4'-dimethoxybenzhydrol (2.57 g, 10.5 mmol) according to the general procedure. An additional portion of NaH (120 mg, 5.0 mmol) was added because the cyclization had not proceeded to completion. Purification by silica gel column chromatography using 10:1 hexane:Et_2O as the eluent gave the product as an off-white solid (2.50 g, 57% yield). **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.55–7.49 (m, 2H), 7.28–7.22 (m, 2H), 7.08 (t, J = 8.0 Hz, 1H), 6.91–6.86 (m, 2H), 6.82 (d, J = 4.6 Hz, 1H), 6.81–6.77 (m, 3H), 4.83 (s, 1H), 3.79 (s, 6H), 0.80 (s, 9H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 159.74, 159.35, 159.08, 134.81, 132.70, 130.55, 130.29, 130.23, 128.58, 121.99, 113.62, 112.25, 109.17, 96.03, 63.07, 55.39, 55.33, 51.18, 30.26; **HRMS** (FAB+) *m/z* Calcd for $C_{26}H_{28}ClNO_3$ $[M]^+$ 437.1758, found 437.1738.



4r

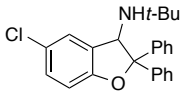
***N*-(*tert*-butyl)-4-chloro-2,2-bis(4-fluorophenyl)-2,3-dihydrobenzofuran-3-amine (4r):** The title compound was prepared from 2-chloro-6-fluorobenzaldehyde (1.59 g, 10.0 mmol), *tert*-butylamine (1.04 mL, 10.0 mmol), and 4,4'-difluorobenzhydrol (2.31 g, 10.5 mmol) according to the general procedure. An additional portion of NaH (120 mg, 5.0 mmol) was added because the cyclization had not proceeded to completion. Purification by silica gel column chromatography using a gradient solvent system (100:1 hexane:Et_2O → 40:1 hexane:Et_2O) as the eluent gave the product as a white solid (1.97 g, 48% yield). **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.63–7.53 (m, 2H), 7.33–7.26 (m, 2H), 7.11 (t, J = 8.0 Hz, 1H), 7.09–7.03 (m, 2H), 6.99–6.93 (m, 2H), 6.87–6.80 (m, 2H), 4.81 (s, 1H), 0.79 (s, 9H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 163.55 (d, J_{C-F} = 38.5 Hz), 161.58 (d, J_{C-F} = 38.6 Hz), 159.50, 138.11 (d, J_{C-F} = 3.1 Hz), 135.72 (d, J_{C-F} = 3.0 Hz), 132.10 (d, J_{C-F} = 8.0 Hz), 130.56, 130.54, 129.71, 129.13 (d, J_{C-F} = 8.1 Hz), 122.38, 115.38 (d, J_{C-F} = 21.3 Hz), 113.68 (d, J_{C-F} = 21.1 Hz), 109.28, 95.58, 63.35, 51.17, 30.22; **^{19}F NMR** (282 MHz, $CDCl_3$) δ –114.30 (tt, J_{F-H1} = 8.5 Hz, J_{F-H2} = 5.3 Hz, 1F), –114.78 (tt, J_{F-H1} = 8.6 Hz, J_{F-H2} = 5.5 Hz, 1F); **HRMS** (FAB+) *m/z* Calcd for $C_{24}H_{22}ClF_2NO$ $[M]^+$ 413.1358, found 413.1345.



4s

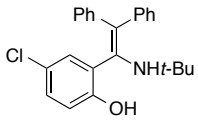
***N*-(*tert*-butyl)-4-chloro-10',11'-dihydro-3*H*-spiro[benzofuran-2,5'-dibenzo[*a*,*d*][7]annulen]-3-amine (4s):** The title compound was prepared from 2-chloro-6-fluorobenzaldehyde (1.59 g, 10.0 mmol), *tert*-butylamine (1.04 mL, 10.0 mmol), and dibenzosuberol (2.21 g, 10.5 mmol) according to the general procedure. Purification by silica gel column chromatography using a gradient solvent system (100:1 hexane:Et_2O → 40:1 hexane:Et_2O) as the eluent gave the product as a white solid (2.67 g, 66% yield). **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.90 (dd, J_1 = 7.9 Hz, J_2 = 1.5 Hz, 1H), 7.31 (dd, J_1 = 8.0 Hz, J_2 = 1.2 Hz, 1H), 7.24–7.05 (m, 6H), 7.01 (d, J = 8.0 Hz, 1H), 6.99–6.94 (m, 1H), 6.80 (dd, J_1 = 7.9 Hz, J_2 = 0.8 Hz, 1H), 4.78 (s, 1H),

3.77 (ddd, J_1 = 15.0 Hz, J_2 = 11.4 Hz, J_3 = 3.8 Hz, 1H), 3.39 (ddd, J_1 = 16.6 Hz, J_2 = 6.7 Hz, J_3 = 3.7 Hz, 1H), 3.03 (ddd, J_1 = 16.1 Hz, J_2 = 11.4 Hz, J_3 = 4.0 Hz, 1H), 2.94 (ddd, J_1 = 14.9 Hz, J_2 = 6.7 Hz, J_3 = 4.0 Hz, 1H), 0.87 (s, 9H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 159.23, 140.87, 139.75, 138.20, 136.88, 131.22, 130.87, 130.70, 130.23, 129.91, 129.53, 127.80, 127.68, 126.50, 126.20, 125.40, 122.20, 109.03, 97.31, 66.19, 50.65, 34.50, 32.83, 30.65; **HRMS** (FAB+) *m/z* Calcd for $C_{26}H_{26}ClNO$ $[M]^+$ 403.1703, found 403.1716.



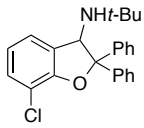
4t

***N*-(*tert*-butyl)-5-chloro-2,2-diphenyl-2,3-dihydrobenzofuran-3-amine (4t):** The title compound was prepared from 5-chloro-2-fluorobenzaldehyde (1.59 g, 10.0 mmol), *tert*-butylamine (1.04 mL, 10.0 mmol), and diphenylmethanol (1.93 g, 10.5 mmol) according to the general procedure. An additional portion of NaH (120 mg, 5.0 mmol) was added because the cyclization had not proceeded to completion. Purification by silica gel column chromatography using a gradient solvent system (100:1 hexane:Et_2O → 40:1 hexane:Et_2O) as the eluent provided a mixture of **4t** and **6t** in 1.3:1 molar ratio, as determined by ^{1}H NMR (2.78 g, 74% yield). Analytically pure samples of both **4t** and **6t** were obtained for the purposes of characterization via PTLC using 100:1 hexane:Et_2O as the eluent. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.67–7.61 (m, 2H), 7.41–7.32 (m, 4H), 7.32–7.23 (m, 4H), 7.17 (d, J = 2.2 Hz, 1H), 7.10 (dd, J_1 = 8.5 Hz, J_2 = 2.2 Hz, 1H), 6.86 (d, J = 8.5 Hz, 1H), 4.88 (s, 1H), 1.24 (s, 1H), 0.93 (s, 9H); **^{13}C NMR** (100 MHz, $(CD_3)_2CO$) δ 158.22, 144.48, 141.26, 136.06, 129.97, 129.57, 129.08, 128.70, 128.33, 128.22, 127.78, 126.48, 126.02, 112.59, 97.89, 64.13, 51.70, 30.60; **HRMS** (FAB+) *m/z* Calcd for $C_{24}H_{24}ClNO$ $[M]^+$ 377.1546, found 377.1544.



6t

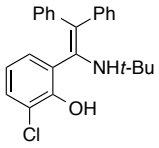
2-(1-(*tert*-butylamino)-2,2-diphenylvinyl)-4-chlorophenol (6t): The title compound was prepared using the procedure described above for **4t**. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 9.06 (s, 1H), 7.44–7.37 (m, 2H), 7.35–7.27 (m, 3H), 7.07–6.98 (m, 4H), 6.90–6.86 (m, 2H), 6.80 (d, J = 2.6 Hz, 1H), 6.77 (d, J = 8.6 Hz, 1H), 3.55 (s, 1H), 1.04 (s, 9H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 155.54, 140.98, 140.52, 137.46, 130.85, 130.20, 130.05, 129.28, 129.09, 128.57, 127.89, 127.49, 126.16, 125.82, 124.06, 116.95, 53.92, 30.12; **HRMS** (FAB+) *m/z* Calcd for $C_{24}H_{24}ClNO$ $[M]^+$ 377.1546, found 377.1546.



4u

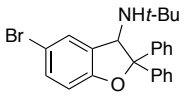
***N*-(*tert*-butyl)-7-chloro-2,2-diphenyl-2,3-dihydrobenzofuran-3-amine (4u):** The title compound was prepared from 3-chloro-2-fluorobenzaldehyde (1.59 g, 10.0 mmol), *tert*-butylamine (1.04 mL, 10.0 mmol), and diphenylmethanol (1.93 g, 10.5 mmol) according to the general procedure. An additional portion of NaH (240 mg, 10.0 mmol) was added because the cyclization had not proceeded to completion. Purification by silica gel column chromatography using a gradient solvent system (40:1 hexane:Et_2O → 20:1 hexane:Et_2O) as the eluent provided a mixture of **4u** and **6u** in 1.7:1 molar ratio, as determined by ^{1}H NMR (2.80 g, 74% yield). Analytically pure samples of both **4u** and **6u** were obtained for the purposes of characterization via PTLC using 100:1 hexane:Et_2O as the eluent. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.72–7.67 (m, 2H), 7.45–7.41 (m, 2H), 7.38–7.33 (m, 2H), 7.31–7.23 (m, 4H), 7.15 (dd, J_1 = 8.0 Hz, J_2 = 1.0 Hz, 1H), 7.11 (dt, J_1 = 7.5 Hz, J_2 = 1.0

Hz, 1H), 6.80 (t, J = 7.7 Hz, 1H), 4.94 (s, 1H), 0.93 (s, 9H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 154.51, 143.20, 139.45, 134.41, 129.18, 129.06, 128.33, 127.94, 127.64, 127.24, 127.20, 123.62, 122.29, 116.16, 96.94, 64.29, 51.17, 30.46; **HRMS** (FAB+) *m/z* Calcd for $C_{24}H_{24}ClNO$ $[M]^+$ 377.1546, found 377.1531.



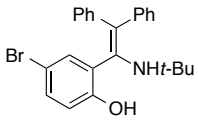
6u

2-(1-(*tert*-butylamino)-2,2-diphenylvinyl)-6-chlorophenol (6u): The title compound was prepared using the procedure described above for **4u**. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 9.79 (s, 1H), 7.43–7.38 (m, 2H), 7.36–7.28 (m, 3H), 7.17 (dd, J_1 = 8.0 Hz, J_2 = 1.6 Hz, 1H), 7.05–6.96 (m, 3H), 6.91–6.85 (m, 3H), 6.73 (dd, J_1 = 7.6 Hz, J_2 = 1.6 Hz, 1H), 6.47 (t, J = 7.8 Hz, 1H), 3.58 (s, 1H), 1.59 (s, 1H), 1.03 (s, 9H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 152.75, 141.16, 140.50, 137.72, 130.29, 130.06, 129.85, 129.76, 129.12, 128.94, 127.84, 127.53, 125.99, 125.83, 120.61, 119.83, 54.04, 30.01; **HRMS** (FAB+) *m/z* Calcd for $C_{24}H_{24}ClNO$ $[M]^+$ 377.1546, found 377.1559; **X-ray** (single-crystal) Colorless block crystals suitable for X-ray diffraction were grown from a solution of the title compound in 40:1 hexane:Et_2O at room temperature (CCDC 1058168).[3]



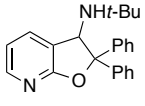
4v

5-bromo-*N*-(*tert*-butyl)-2,2-diphenyl-2,3-dihydrobenzofuran-3-amine (4v): The title compound was prepared from 5-bromo-2-fluorobenzaldehyde (1.18 mL, 10.0 mmol), *tert*-butylamine (1.04 mL, 10.0 mmol), and diphenylmethanol (1.93 g, 10.5 mmol) according to the general procedure. An additional portion of NaH (192 mg, 8.0 mmol) was added because the cyclization had not proceeded to completion. Purification by silica gel column chromatography using 40:1 hexane:Et_2O as the eluent provided a mixture of **4v** and **6v** in 6.3:1 molar ratio, as determined by ^{1}H NMR (3.67 g, 87% yield). Analytically pure samples of both **4v** and **6v** were obtained for the purposes of characterization via PTLC using 100:1 hexane:Et_2O as the eluent. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.67–7.64 (m, 2H), 7.42–7.25 (m, 10H), 6.85 (d, J = 8.5 Hz, 1H), 4.91 (s, 1H), 0.95 (s, 9H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 157.52, 143.22, 139.70, 135.05, 131.87, 129.00, 128.37, 128.30, 127.96, 127.66, 127.33, 127.22, 113.00, 112.36, 96.75, 63.34, 51.18, 30.47; **HRMS** (FAB+) *m/z* Calcd for $C_{24}H_{24}{}^{81}BrNO$ $[M]^+$ 423.1021, found 423.1033.



6v

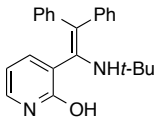
4-bromo-2-(1-(*tert*-butylamino)-2,2-diphenylvinyl)phenol (6v): The title compound was prepared using the procedure described above for **4v**. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 9.10 (s, 1H), 7.40 (t, J = 7.5 Hz, 2H), 7.35–7.28 (m, 3H), 7.17 (dd, J_1 = 8.6 Hz, J_2 = 2.5 Hz, 1H), 7.10–6.99 (m, 3H), 6.93 (d, J = 2.5 Hz, 1H), 6.90–6.86 (m, 2H), 6.72 (d, J = 8.6 Hz, 1H), 3.54 (s, 1H), 1.04 (s, 9H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 156.04, 140.96, 140.49, 137.36, 133.77, 132.17, 130.21, 130.04, 129.10, 128.71, 127.90, 127.50, 126.36, 126.19, 117.45, 111.18, 53.95, 30.14; **HRMS** (FAB+) *m/z* Calcd for $C_{24}H_{24}{}^{81}BrNO$ $[M]^+$ 423.1021, found 423.1019.



4w

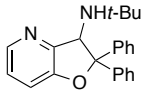
***N*-(*tert*-butyl)-2,2-diphenyl-2,3-dihydrofuro[2,3-b]pyridin-3-amine (4w):** The title compound was prepared from 2-fluoro-3-formylpyridine (1.25 g, 10.0 mmol), *tert*-butylamine (1.04 mL, 10.0 mmol), and diphenylmethanol (1.93 g, 10.5 mmol)

according to the general procedure. Purification by silica gel column chromatography using a gradient solvent system (80:20:1 DCM:hexane:methanol → 25:1 DCM:methanol) as the eluent provided **4w** as a brown oil (497 mg, 14% yield) and **6w** as a brown solid (113 mg, 3% yield). **^{1}H NMR** (500 MHz, $CDCl_3$) δ 8.08 (dd, J_1 = 5.1 Hz, J_2 = 1.4 Hz, 1H), 7.74–7.70 (m, 2H), 7.53 (ddd, J_1 = 7.2 Hz, J_2 = 1.7 Hz, J_3 = 0.8 Hz, 1H), 7.46–7.42 (m, 2H), 7.37–7.32 (m, 2H), 7.31–7.22 (m, 4H), 6.81 (dd, J_1 = 7.2, J_2 = 5.1 Hz, 1H), 4.98 (s, 1H), 0.98 (s, 9H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 166.73, 147.98, 143.25, 139.48, 134.45, 128.53, 128.31, 127.97, 127.63, 127.37, 127.23, 125.56, 117.60, 94.69, 62.06, 51.22, 30.59; **HRMS** (FAB+) *m/z* Calcd for $C_{23}H_{24}N_2O$ $[M]^+$ 344.1889, found 344.1878.



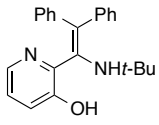
6w

3-(1-(*tert*-butylamino)-2,2-diphenylvinyl)pyridin-2-ol (6w): The title compound was prepared using the procedure described above for **4w**. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 13.06 (s, 1H), 7.44–7.31 (m, 6H), 7.22 (tt, J_1 = 7.1 Hz, J_2 = 2.0 Hz, 1H), 7.05–6.97 (m, 4H), 6.95–6.89 (m, 1H), 6.09 (t, J = 6.6 Hz, 1H), 3.85 (s, 1H), 1.11 (s, 9H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 164.78, 143.94, 143.30, 141.96, 139.90, 134.99, 130.94, 130.90, 130.21, 128.70, 127.63, 126.51, 125.15, 121.89, 106.47, 52.52, 30.81; **HRMS** (FAB+) *m/z* Calcd for $C_{23}H_{25}N_2O$ $[M+H]^+$ 345.1967, found 345.1959.



4x

***N*-(*tert*-butyl)-2,2-diphenyl-2,3-dihydrofuro[3,2-b]pyridin-3-amine (4x):** The title compound was prepared from 3-fluoro-2-formylpyridine (1.25 g, 10.0 mmol), *tert*-butylamine (1.04 mL, 10.0 mmol), and diphenylmethanol (1.93 g, 10.5 mmol) according to the general procedure. An additional portion of NaH (126 mg, 4.0 mmol) was added because the cyclization had not proceeded to completion. After the first chromatographic purification on silica gel (2:1 hexane:Et_2O), the product still contained unidentifiable impurities. A second purification by silica gel column chromatography (80:20:1 DCM:hexane:methanol → 80:10:1 DCM:hexane:methanol) provided **4x** as a brown oil (804 mg, 23% yield) and **6x** as a white solid (1.11 g, 32%). **^{1}H NMR** (500 MHz, $CDCl_3$) δ 8.10 (d, J = 4.7 Hz, 1H), 7.66 (d, J = 7.7 Hz, 2H), 7.42 (d, J = 7.5 Hz, 2H), 7.37–7.21 (m, 6H), 7.15 (d, J = 8.1 Hz, 1H), 7.07–6.98 (m, 1H), 4.99 (s, 1H), 1.01 (s, 9H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 154.43, 152.18, 143.25, 142.76, 139.72, 128.87, 128.28, 127.99, 127.66, 127.35, 127.29, 123.32, 116.98, 96.04, 63.53, 51.43, 30.42; **HRMS** (FAB+) *m/z* Calcd for $C_{23}H_{24}N_2O$ $[M]^+$ 344.1889, found 344.1886.



6x

2-(1-(*tert*-butylamino)-2,2-diphenylvinyl)pyridin-3-ol (6x): The title compound was prepared using the procedure described above for **4x**. The NMR spectra for **6x** suggest that two rotamers may be present in solution; only peaks corresponding to the major rotamer are reported. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 9.08 (s, 1H), 7.89–7.80 (m, 1H), 7.40 (d, J = 6.5 Hz, 3H), 7.35–7.28 (m, 1H), 7.27–7.21 (m, 1H), 7.14–7.09 (m, 1H), 7.06–6.93 (m, 4H), 6.85–6.78 (m, 2H), 3.64 (s, 1H), 1.05 (s, 9H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 153.54, 144.15, 141.05, 140.85, 140.50, 137.69, 130.16, 129.97, 129.50, 129.02, 127.72, 127.50, 125.74, 124.15, 122.34, 54.24, 30.21; **HRMS** (FAB+) *m/z* Calcd for $C_{23}H_{24}N_2O$ $[M]^+$ 344.1889, found 344.1874.

X-RAY CRYSTALLOGRAPHY METHODS AND RESULTS

Refinement Details: In each case, crystals were mounted on a glass fiber or nylon loop using Paratone oil, then placed on the diffractometer under a nitrogen stream. Low-temperature (100 K) X-ray data were obtained on a Bruker Kappa diffractometer coupled to a Apex II CCD detector with graphite monochromated Mo K_α radiation (λ = 0.71073 Å) for the structures of compounds **4h** (CCDC 1058167) and **4n** (CCDC 1058169) and on a Bruker AXS D8 VENTURE KAPPA diffractometer coupled to a PHOTON 100 CMOS detector with Mo K_α radiation (λ = 0.71073 Å) from an IμS micro-source for the structure of compound **6u** (CCDC 1058168). All diffractometer manipulations, including data collection, integration, and scaling were carried out using the Bruker APEXII software.[4] Absorption corrections were applied using SADABS.[5] Space groups were determined on the basis of systematic absences and intensity statistics and the structures were solved by direct methods using XS[6] or by intrinsic phasing using XT (incorporated into SHELXTL) and refined by full-matrix least squares on F^2. All non-hydrogen and hydride atoms were refined using anisotropic displacement parameters. Non-hydride hydrogen atoms were placed in the idealized positions and refined using a riding model. The structure was refined (weighed least squares refinement on F^2) to convergence. Graphical representation of structures with 50% probability thermal ellipsoids was generated using Diamond visualization software.[7]

Table S2: Crystal data and structure refinement for **4h** (CCDC 1058167).

Identification code	a14314
Empirical formula	C24 H24 Cl N O
Formula weight	377.89
Temperature	100 K
Wavelength	0.71073 Å
Crystal system	Monoclinic
Space group	P 1 21/c 1
Unit cell dimensions	a = 16.3879(9) Å α = 90°
	b = 7.0026(4) Å β = 103.718(3)°
	c = 17.8667(10) Å g = 90°
Volume	1991.86(19) $Å^3$
Z	4
Density (calculated)	1.260 g/cm^3
Absorption coefficient	0.205 mm^{-1}
F(000)	800
Crystal size	0.41 × 0.39 × 0.34 mm^3
Theta range for data collection	2.392 to 48.941°.
Index ranges	-34 ≤ h ≤ 34, -14 ≤ k ≤ 14, -34 ≤ l ≤ 37
Reflections collected	152651
Independent reflections	19792 [R(int) = 0.0448]
Completeness to theta = 25.000°	99.9 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	1.0000 and 0.9478
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	19792 / 0 / 340
Goodness-of-fit on F^2	1.703
Final R indices [I>2σ(I)]	R1 = 0.0440, wR2 = 0.1046
R indices (all data)	R1 = 0.0713, wR2 = 0.1102
Extinction coefficient	n/a
Largest diff. peak and hole	0.609 and -0.689 $e·Å^{-3}$

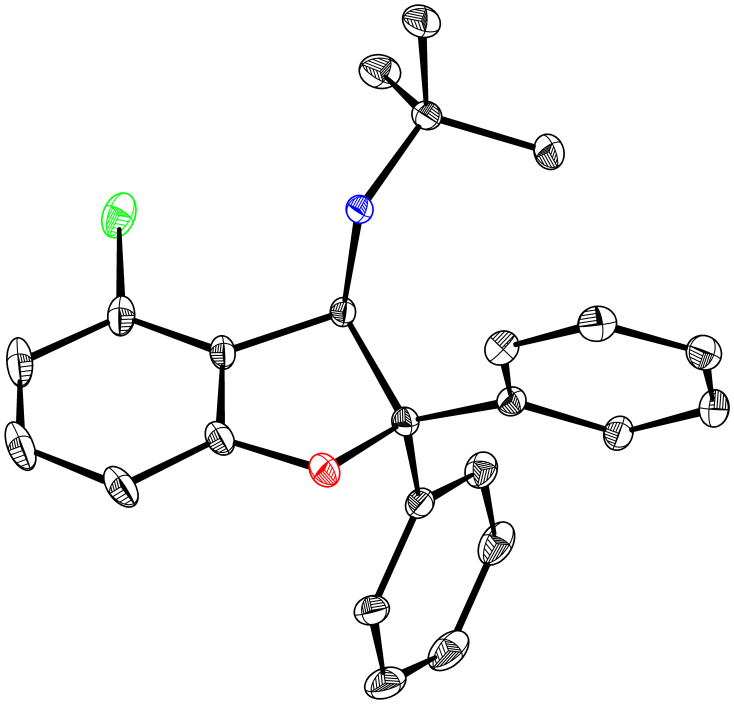


Figure S1. X-ray crystal structure of **4h** with 50% probability ellipsoids. Hydrogen atoms omitted for clarity. CCDC 1058167.

Table S3: Crystal data and structure refinement for **4n** (CCDC 1058169).

Identification code	P14056	
Empirical formula	C39 H30 Cl N O	
Formula weight	564.09	
Temperature	160(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	$P2_1/n$	
Unit cell dimensions	a = 14.7817(5) Å	α = 90°
	b = 10.4931(3) Å	β = 110.0396(11)°
	c = 20.1054(7) Å	γ = 90°
Volume	2929.66(17) $Å^3$	
Z	4	
Density (calculated)	1.279 g/cm^3	
Absorption coefficient	0.163 mm^{-1}	
F(000)	1184	
Crystal size	0.200 × 0.200 × 0.200 mm^3	
Theta range for data collection	2.449 to 30.503°.	
Index ranges	$-21 \le h \le 21$, $-14 \le k \le 14$, $-28 \le l \le 28$	
Reflections collected	118977	
Independent reflections	8923 [R(int) = 0.0530]	
Completeness to theta = 25.242°	99.8 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.7471 and 0.7196	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	8923 / 1 / 382	
Goodness-of-fit on F^2	1.079	
Final R indices [I>2σ(I)]	R1 = 0.0530, wR2 = 0.1360	
R indices (all data)	R1 = 0.0729, wR2 = 0.1495	
Extinction coefficient	n/a	
Largest diff. peak and hole	0.694 and -0.577 $e.Å^{-3}$	

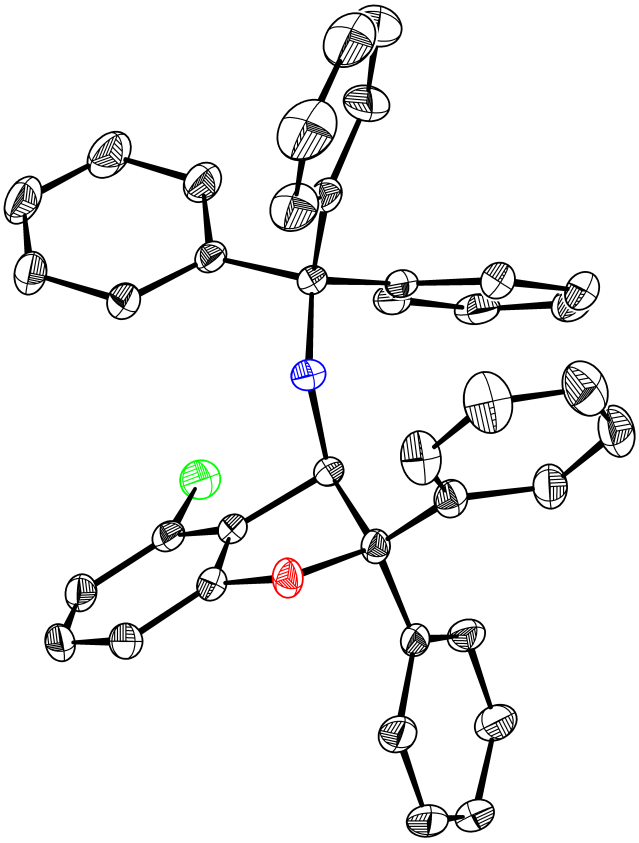


Figure S2. X-ray crystal structure of **4n** with 50% probability ellipsoids. Hydrogen atoms omitted for clarity. CCDC 1058169.

Table S4: Crystal data and structure refinement for **6u** (CCDC 1058168).

Identification code	a14340
Empirical formula	C24 H24 Cl N O
Formula weight	377.89
Temperature	100.0 K
Wavelength	0.71073 Å
Crystal system	Monoclinic
Space group	P 1 21/n 1
Unit cell dimensions	a = 12.0241(5) Å α = 90°
	b = 10.8634(4) Å β = 92.829(2)°
	c = 15.0306(6) Å γ = 90°
Volume	1960.94(13) $Å^3$
Z	4
Density (calculated)	1.280 Mg/m^3
Absorption coefficient	0.208 mm^{-1}
F(000)	800
Crystal size	0.4 x 0.29 x 0.19 mm^3
Theta range for data collection	2.119 to 41.861°.
Index ranges	$-22 \le h \le 22$, $-20 \le k \le 20$, $-28 \le l \le 28$
Reflections collected	140172
Independent reflections	13399 [R(int) = 0.0477]
Completeness to theta = 25.000°	100.0 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	1.0000 and 0.9335
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	13399 / 0 / 340
Goodness-of-fit on F^2	1.047
Final R indices [I>2σ(I)]	R1 = 0.0372, wR2 = 0.1016
R indices (all data)	R1 = 0.0526, wR2 = 0.1120
Extinction coefficient	n/a
Largest diff. peak and hole	0.797 and -0.217 $e.Å^{-3}$

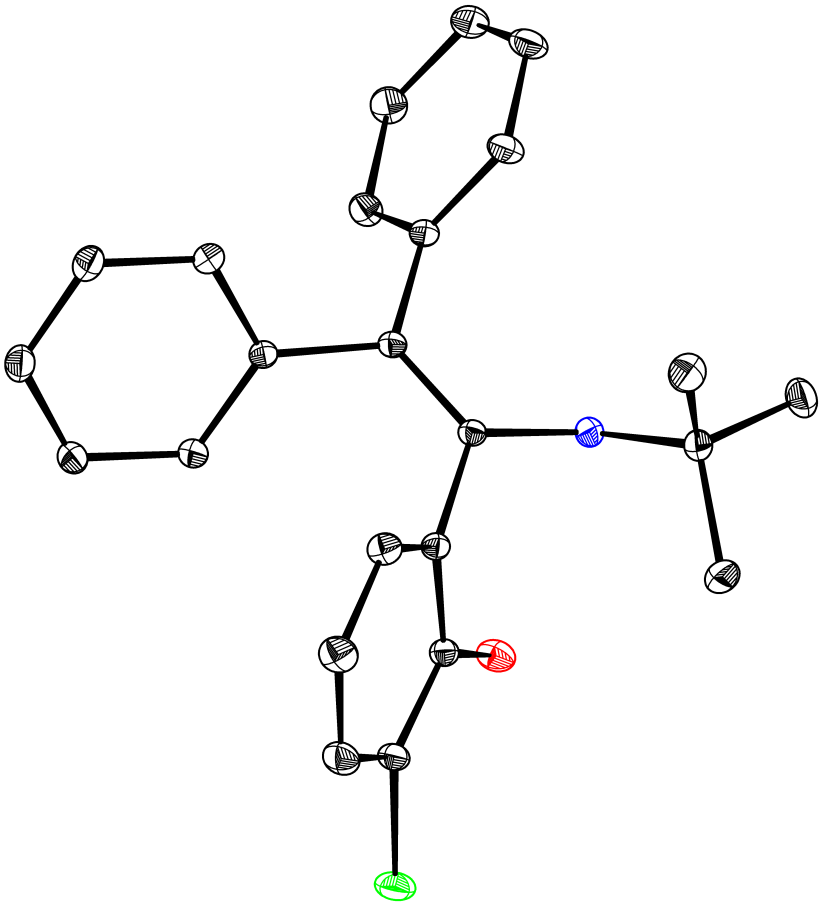


Figure S3. X-ray crystal structure of **6u** with 50% probability ellipsoids. Hydrogen atoms omitted for clarity. CCDC 1058168.

REFERENCES

(1) Franchi, P.; Casati, C.; Mezzina, E.; Lucarini, M. *Org. Biomol. Chem.* **2011**, *9*, 6396–6401.

(2) Forrest, A. K.; Jarvest, R. L.; Sheppard, R. J. (GlaxoSmithKline PLC). Novel Compounds. World Patent WO2007017267 A2, February 15, 2007.

(3) CCDC 1058167 (**4h**), 1058169 (**4n**), and 1058168 (**6u**) contain the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.

(4) *APEX2, Version 2 User Manual*, M86-E01078, Bruker Analytical X-ray Systems, Madison, WI, 2006.

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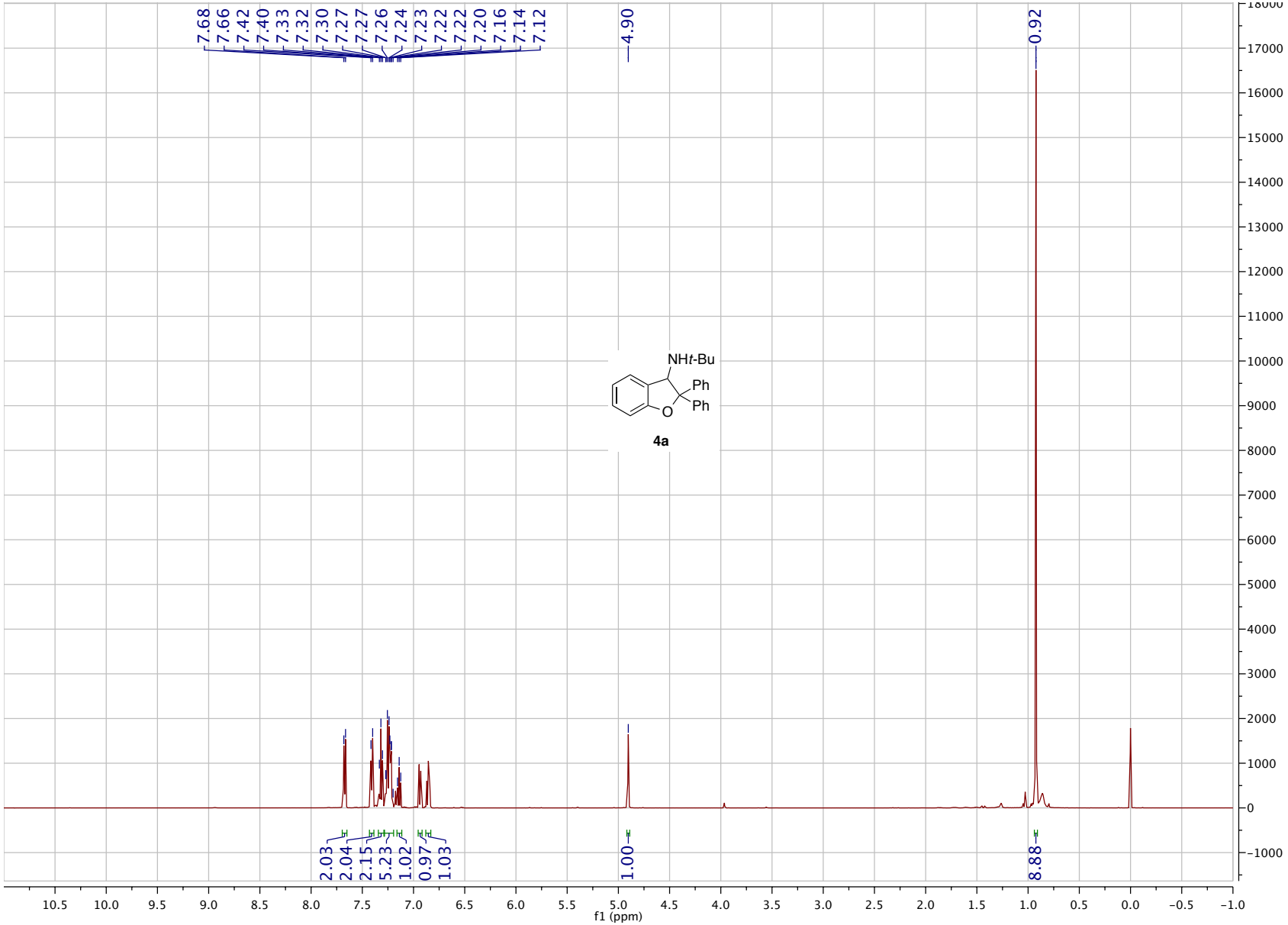
(6) Sheldrick, G. *Acta Crystallogr. Sect. A* **2008**, *64*, 112–122.

(7) (a) Spek, A. L. *PLATON – A Multipurpose Crystallographic Tool*, Utrecht University, Utrecht, The Netherlands, 2006. (b) Spek A. L. *Acta Cryst. Sect. A* **1990**, *46*, C34.

NMR SPECTRA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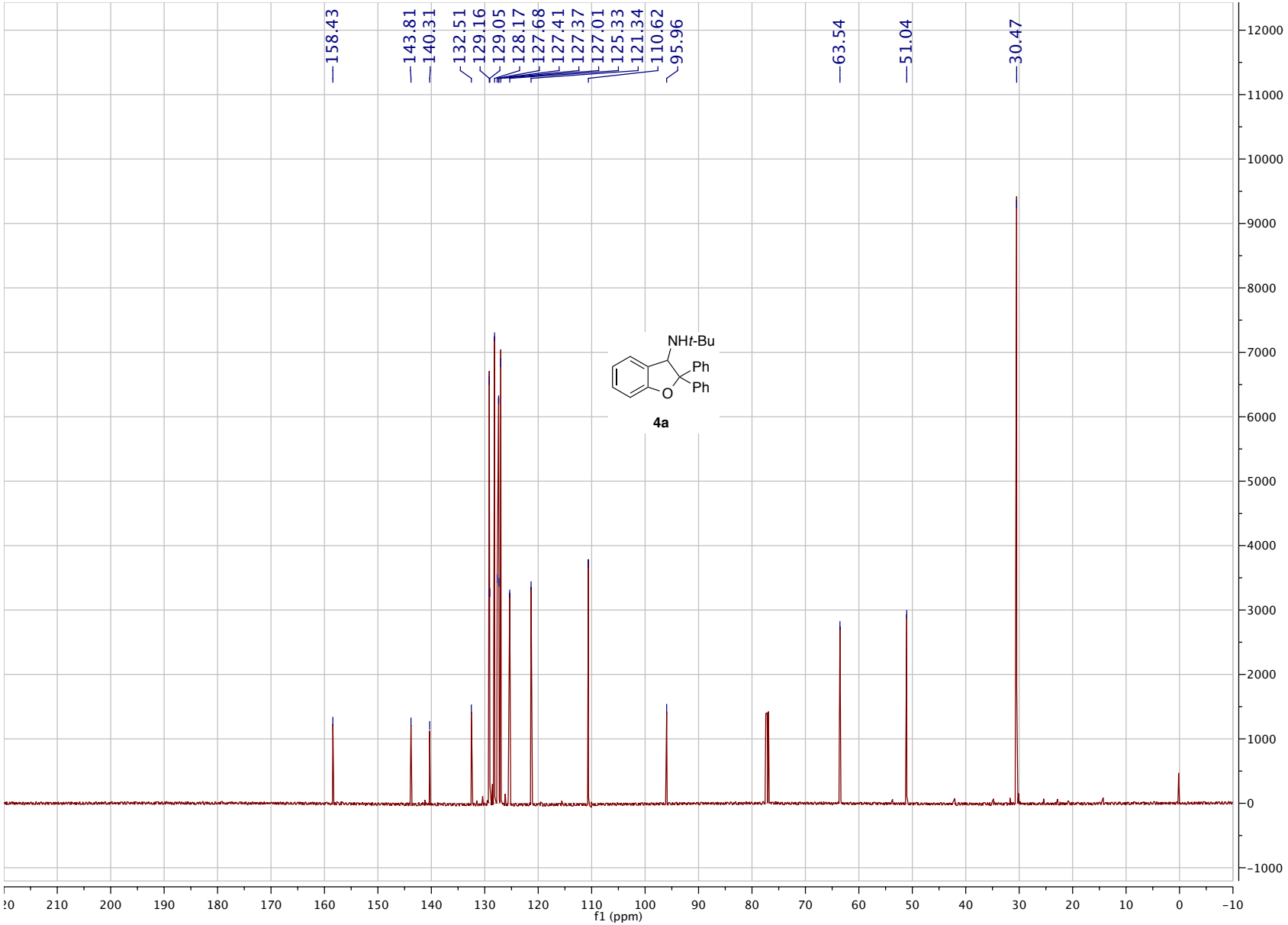
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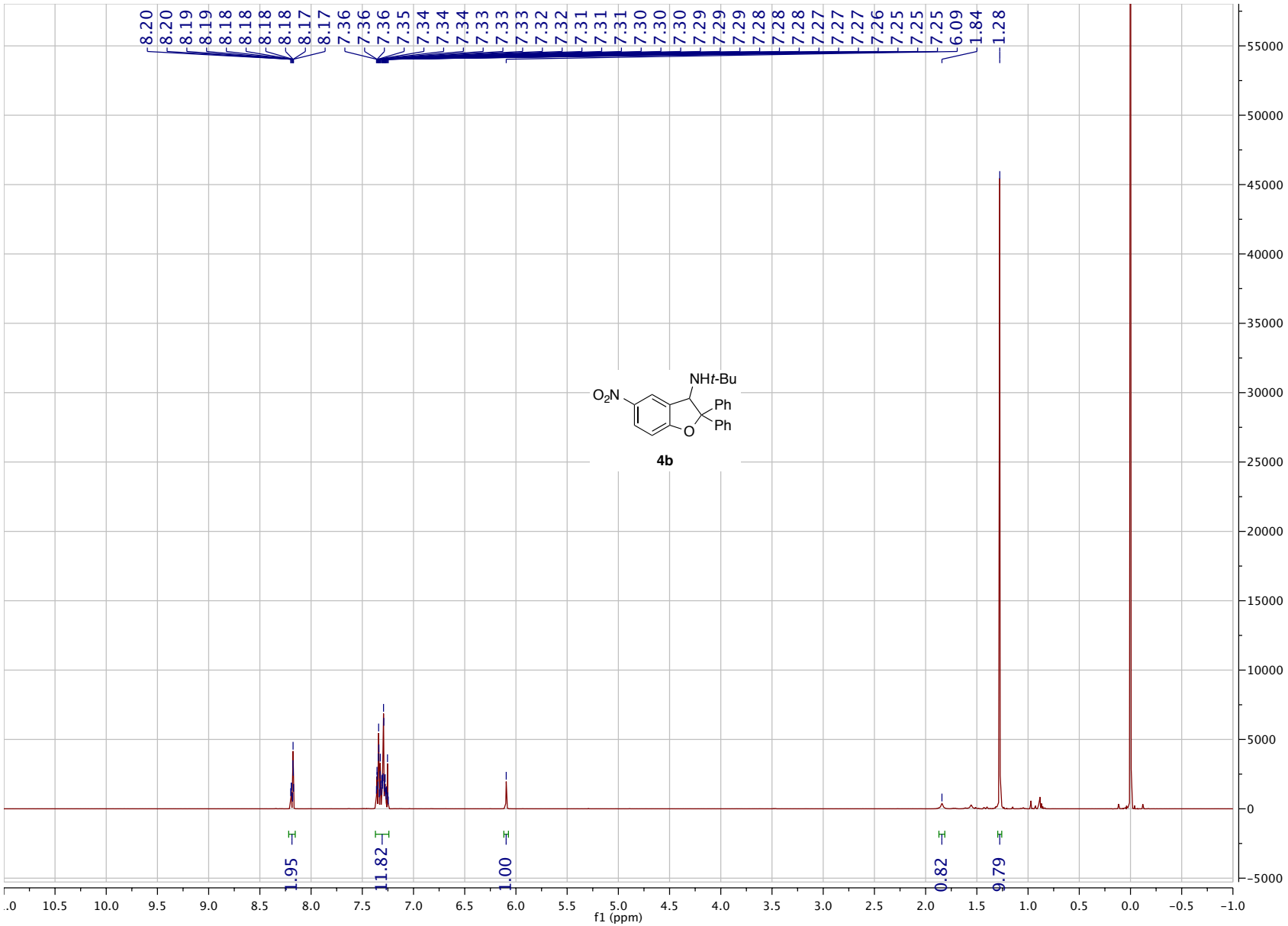
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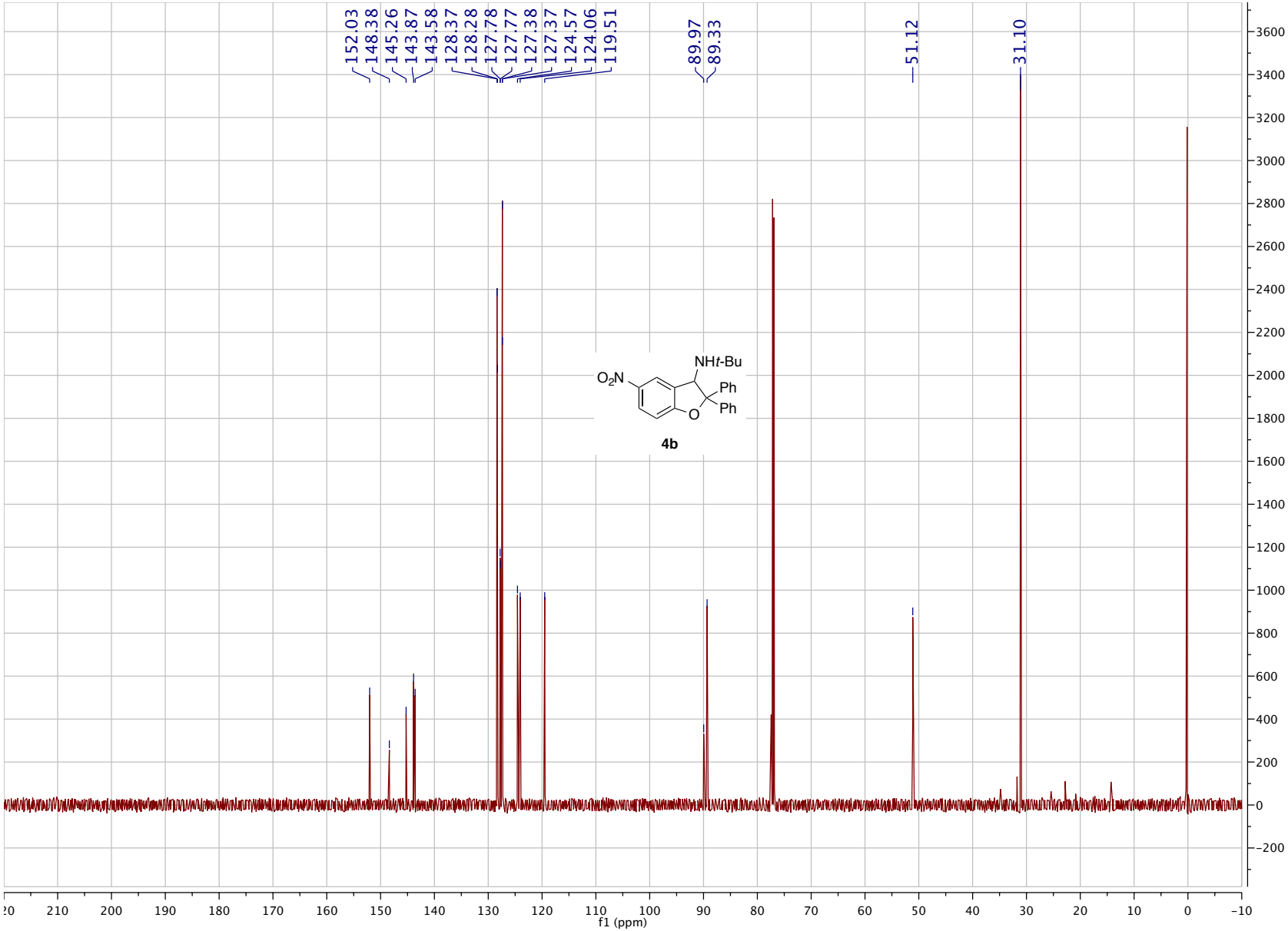
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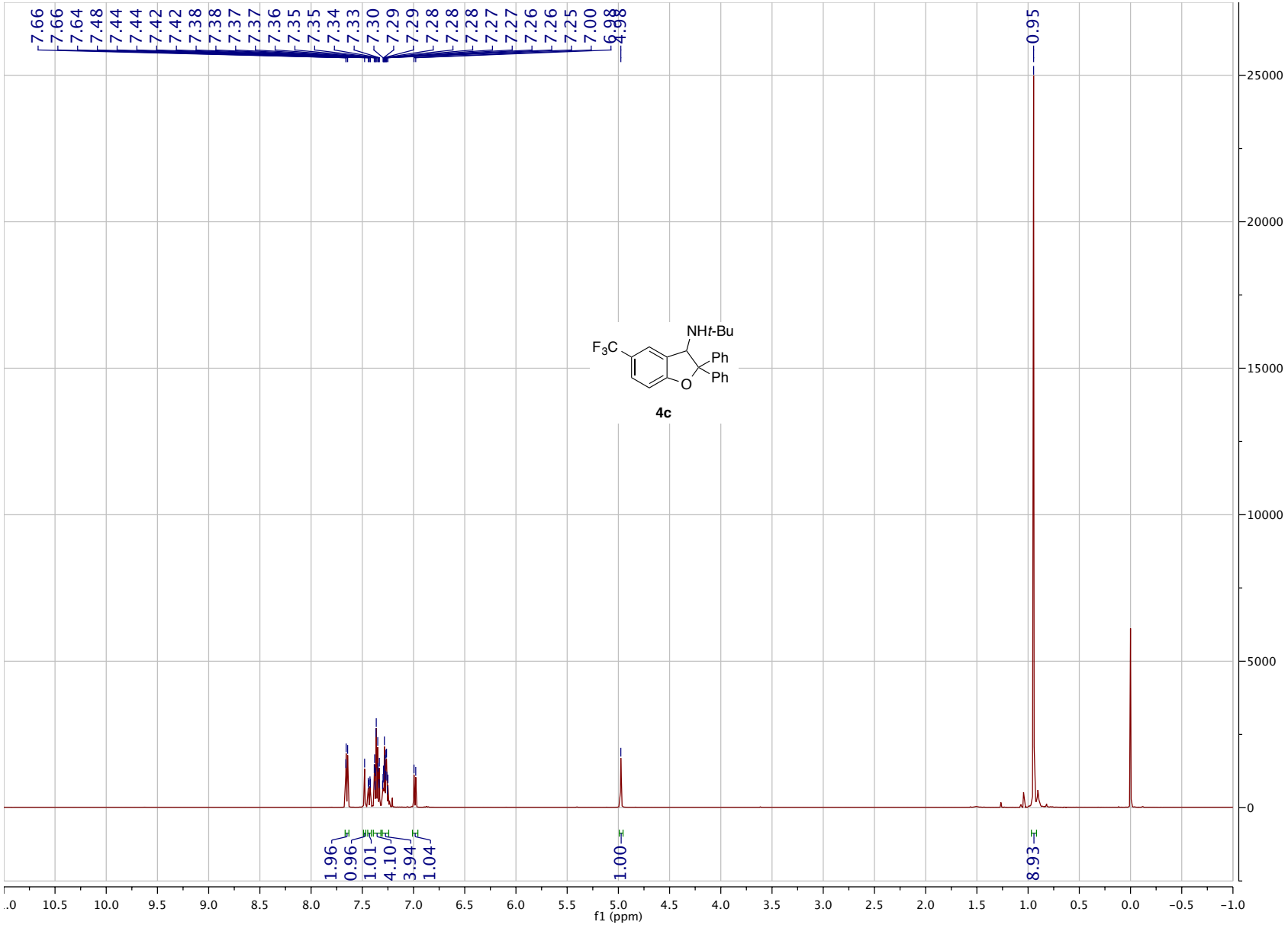
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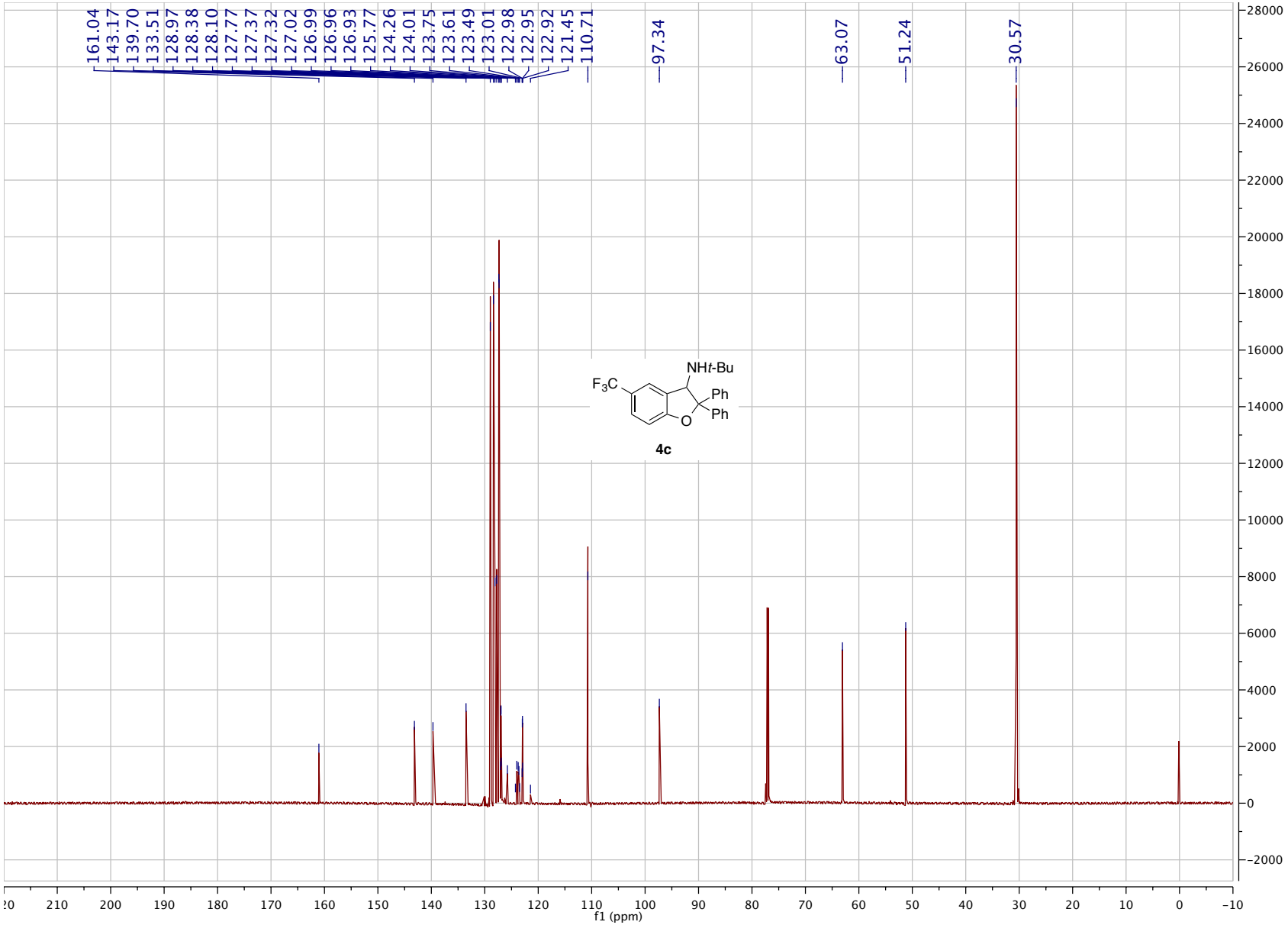
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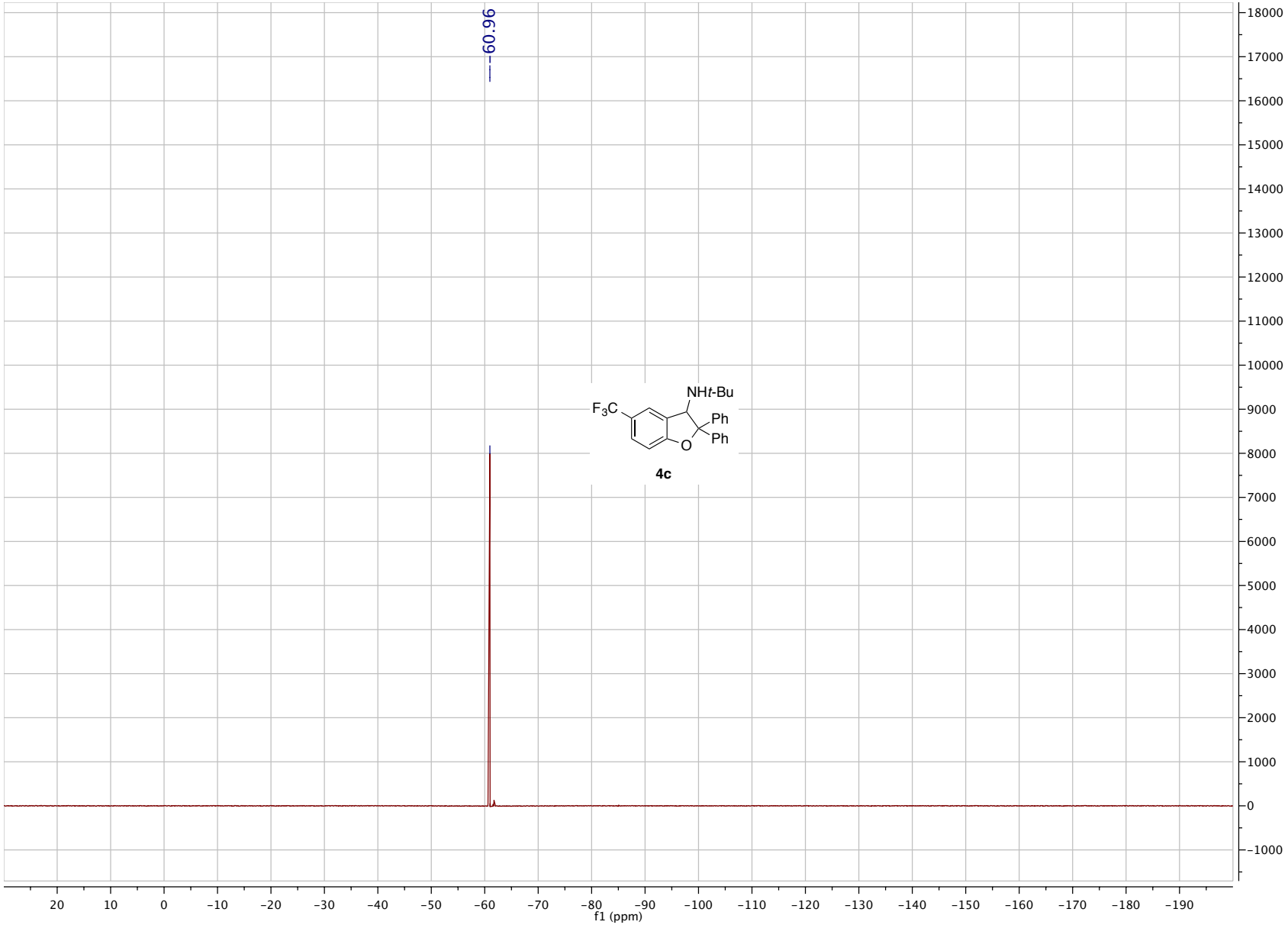
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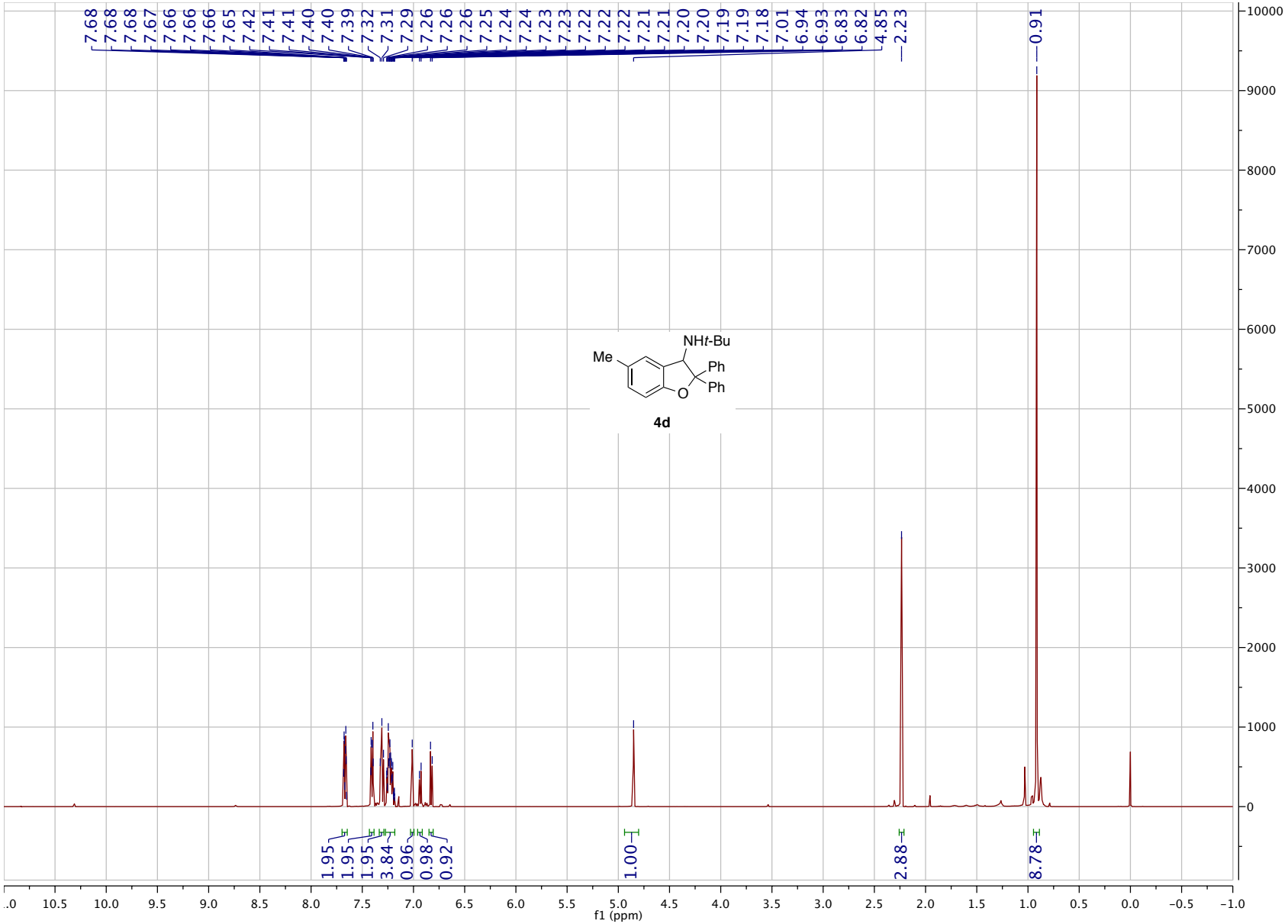
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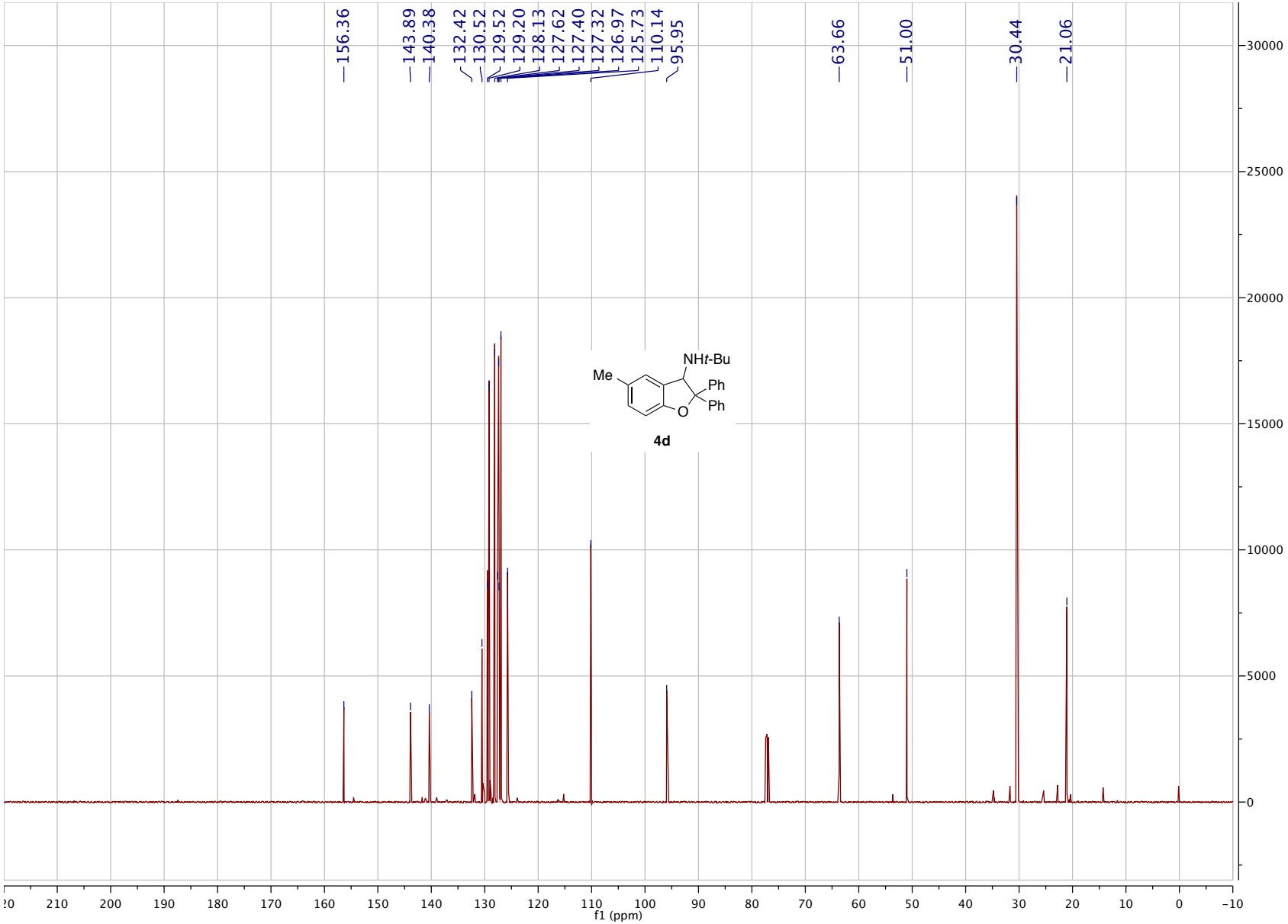
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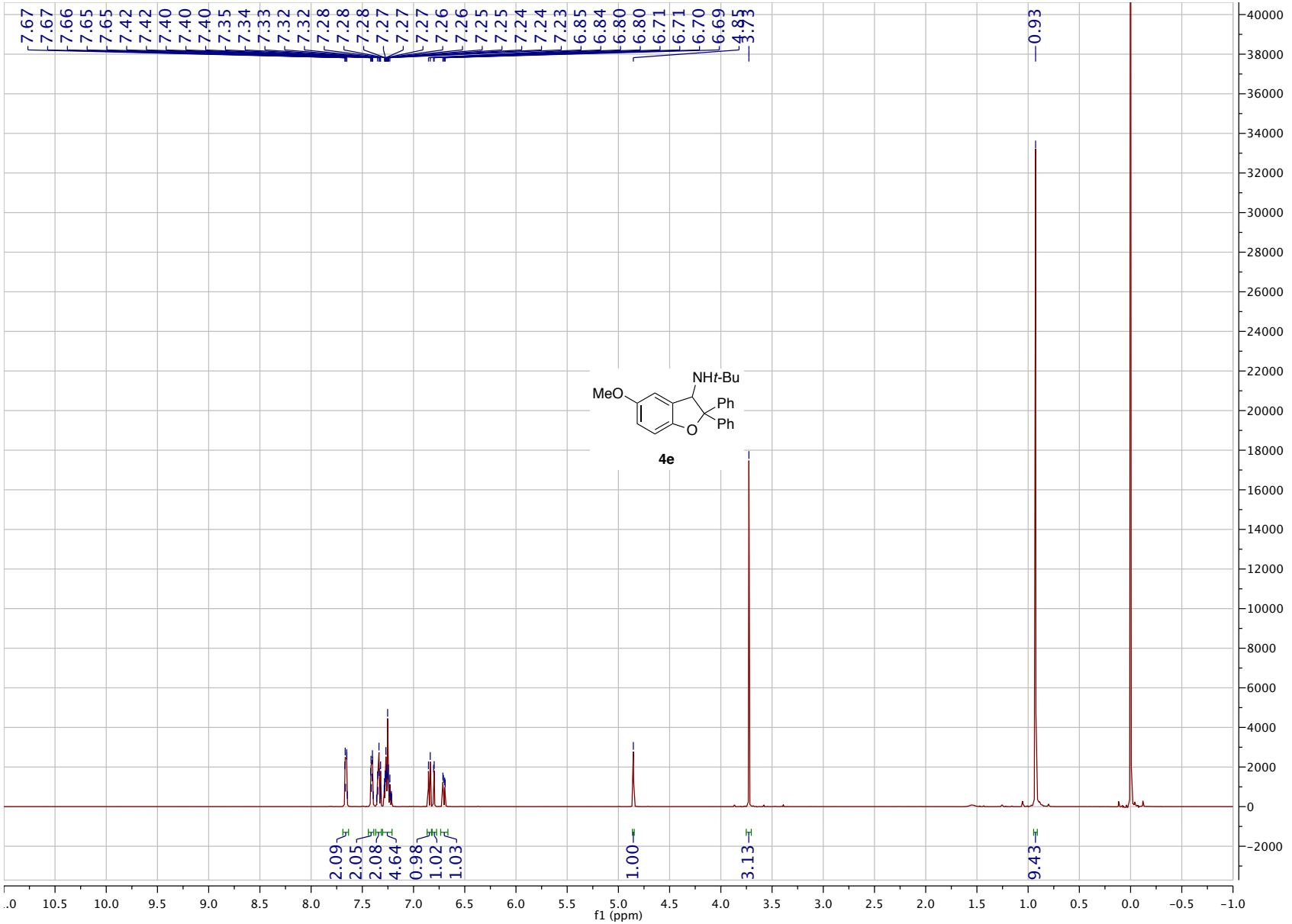
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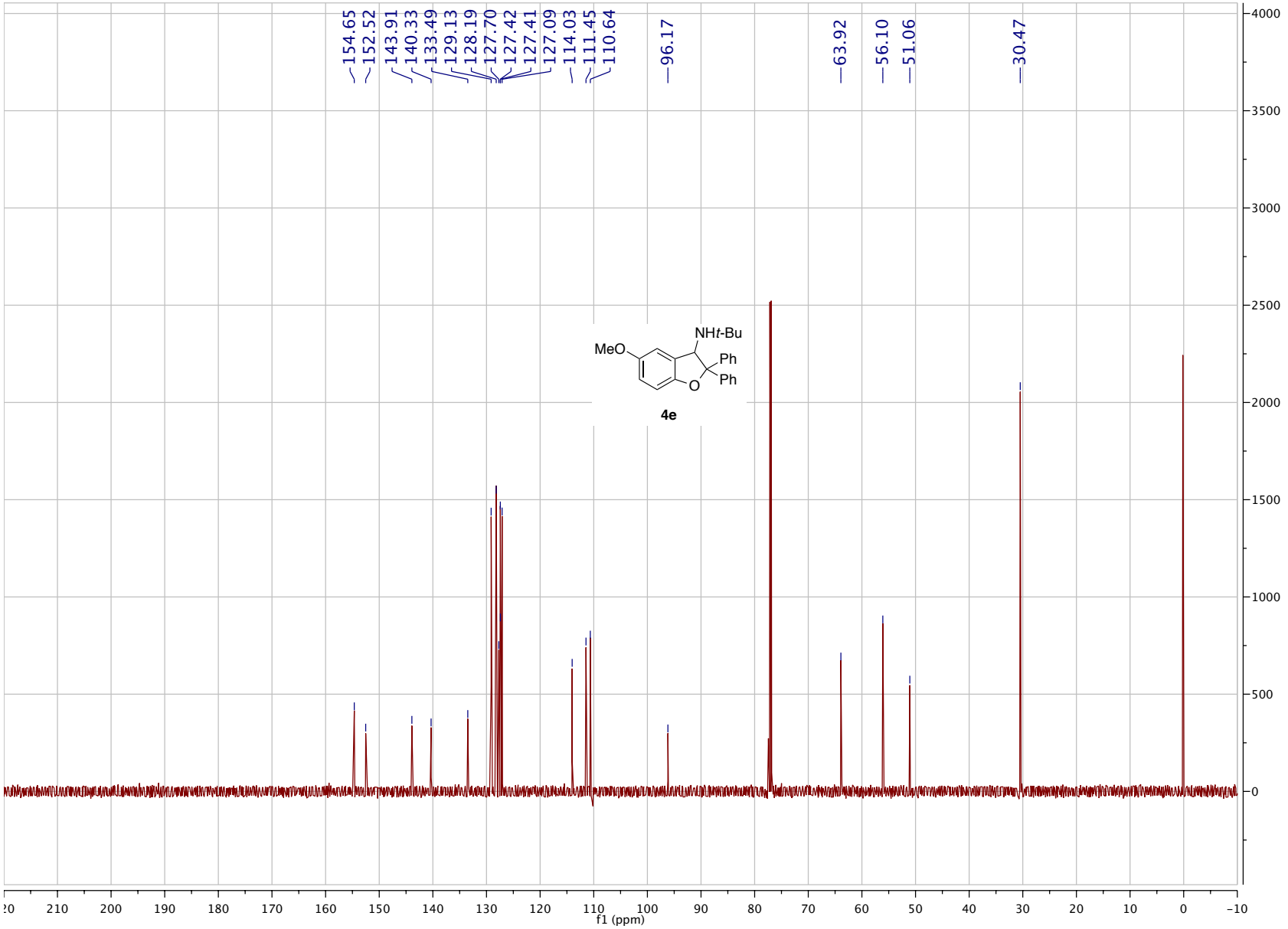
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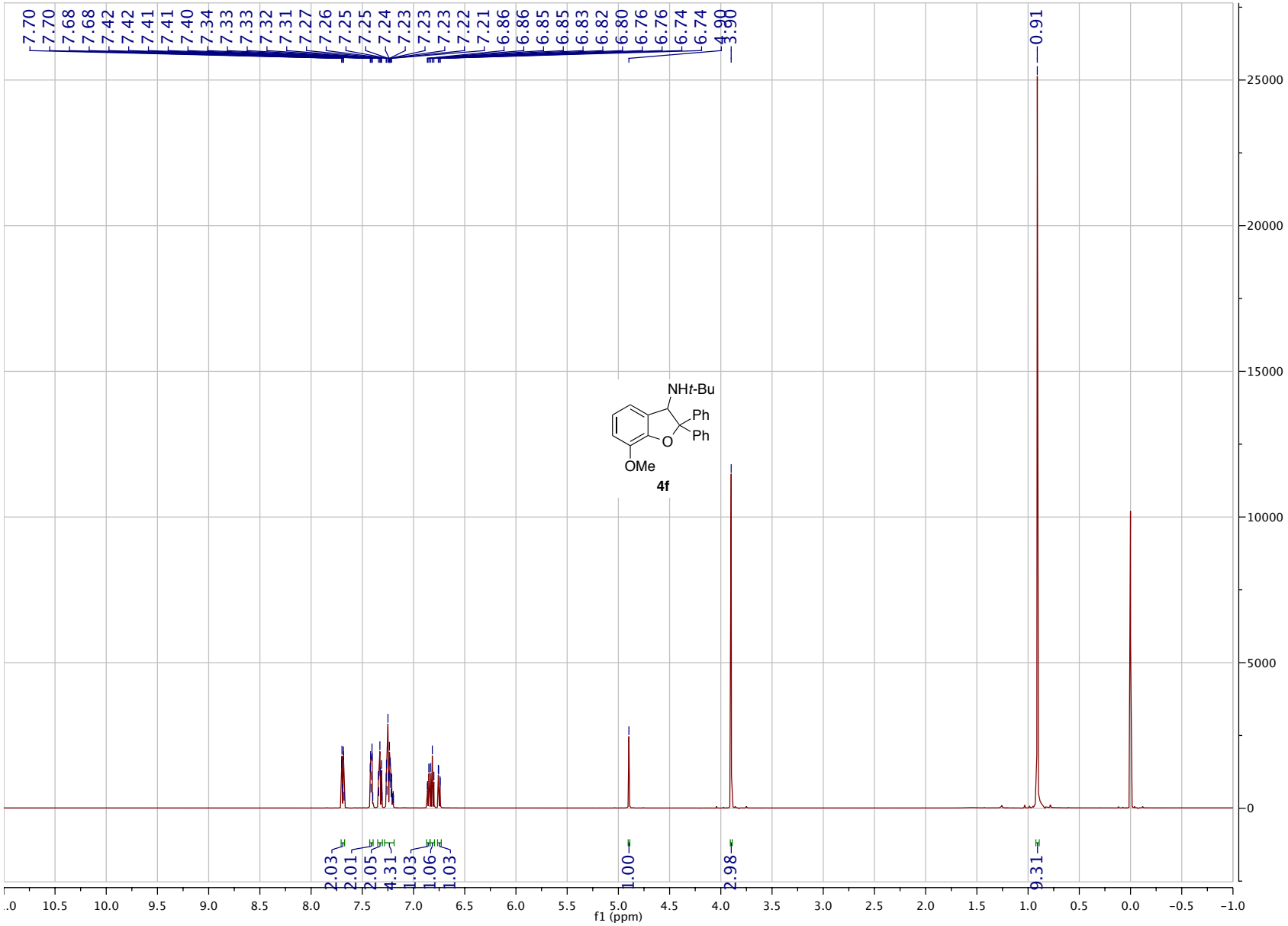
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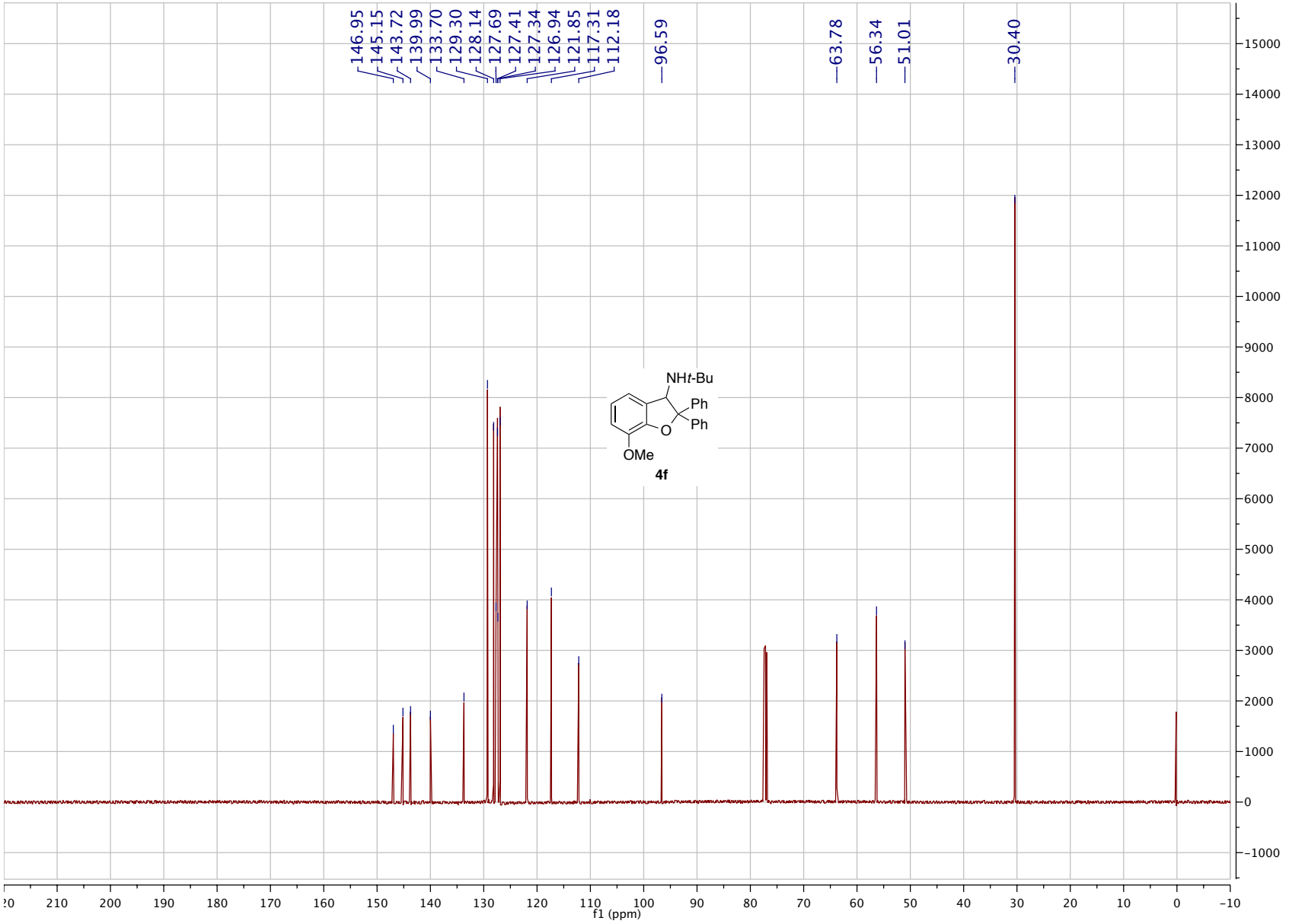
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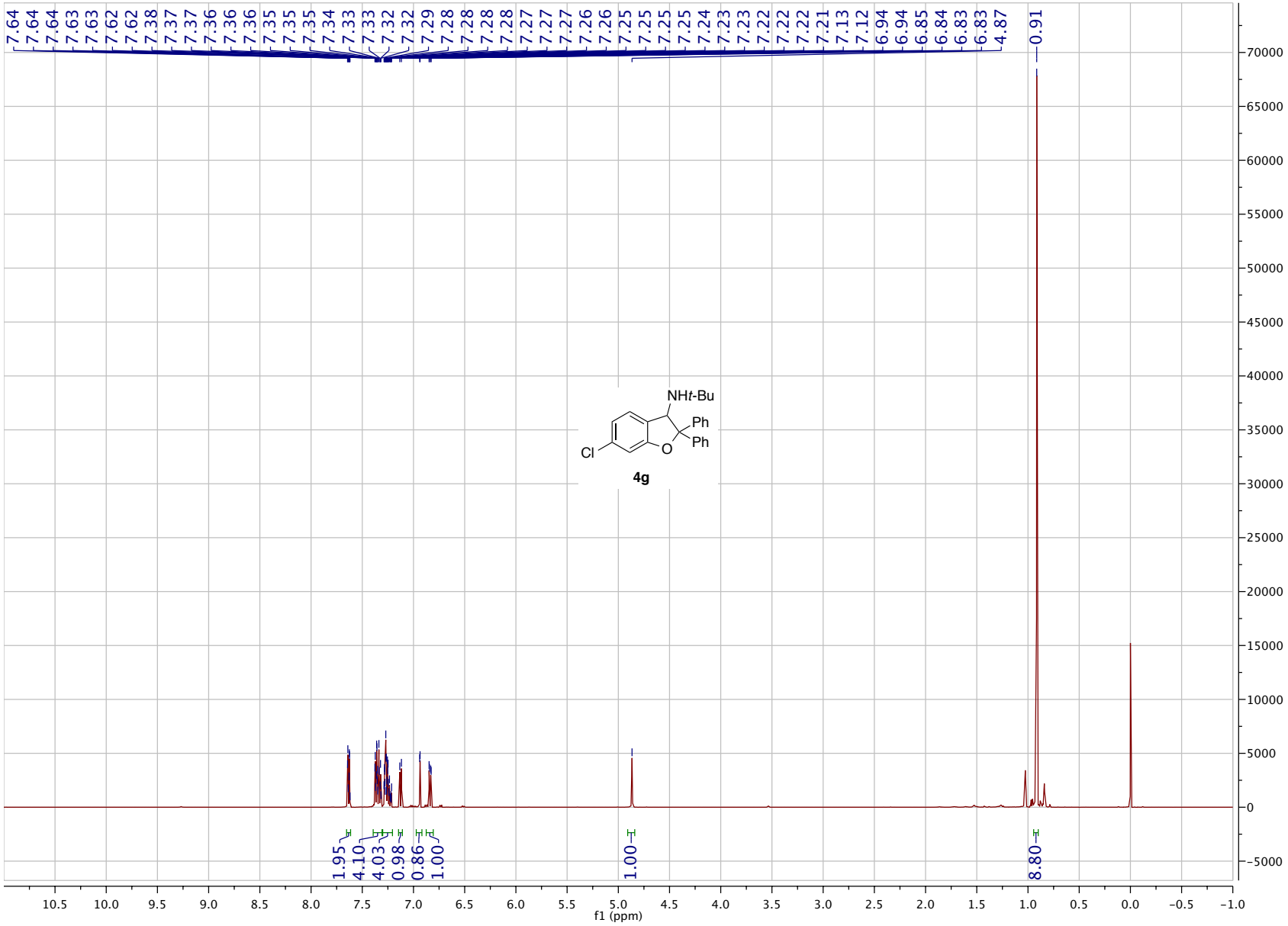
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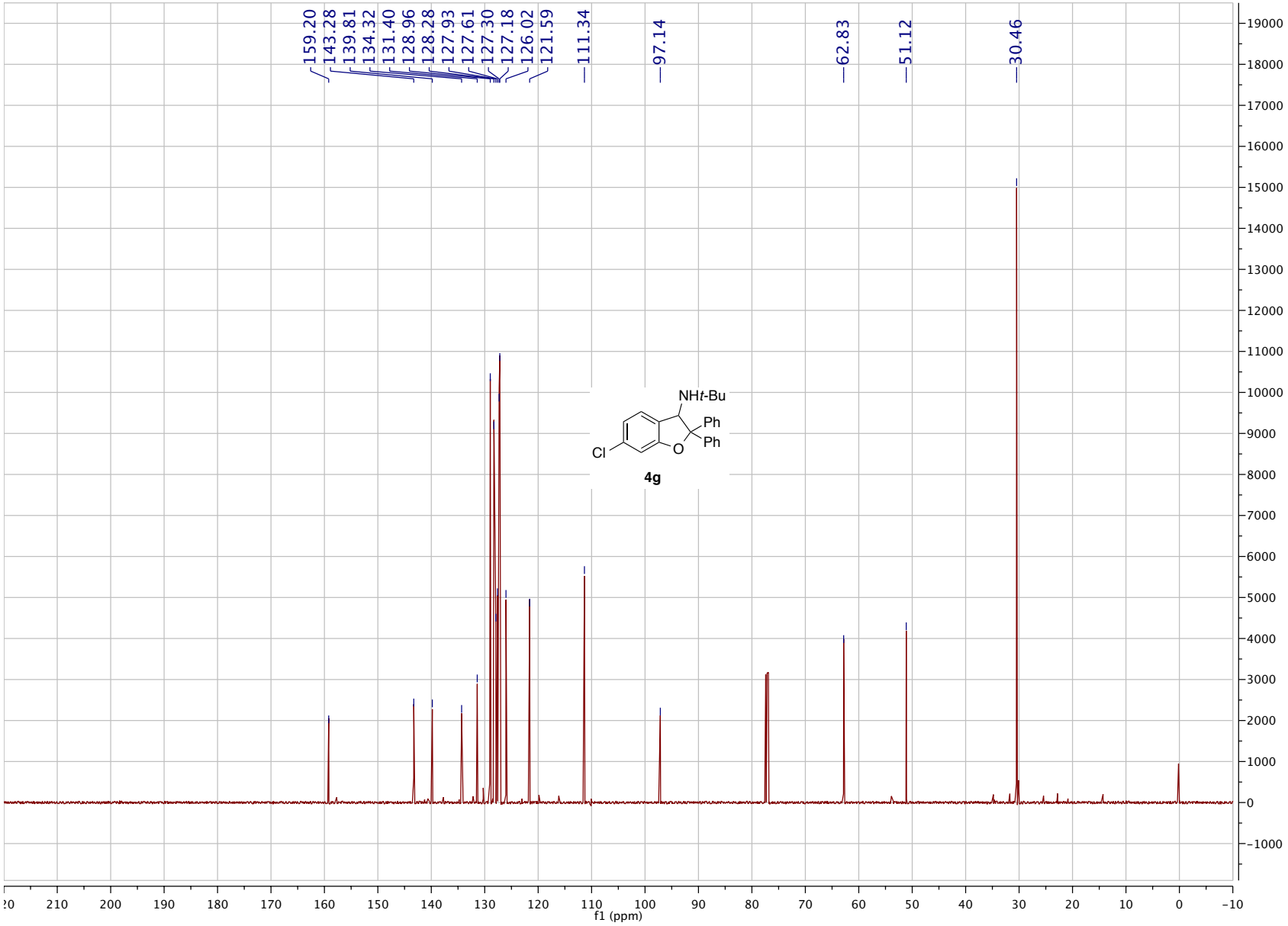
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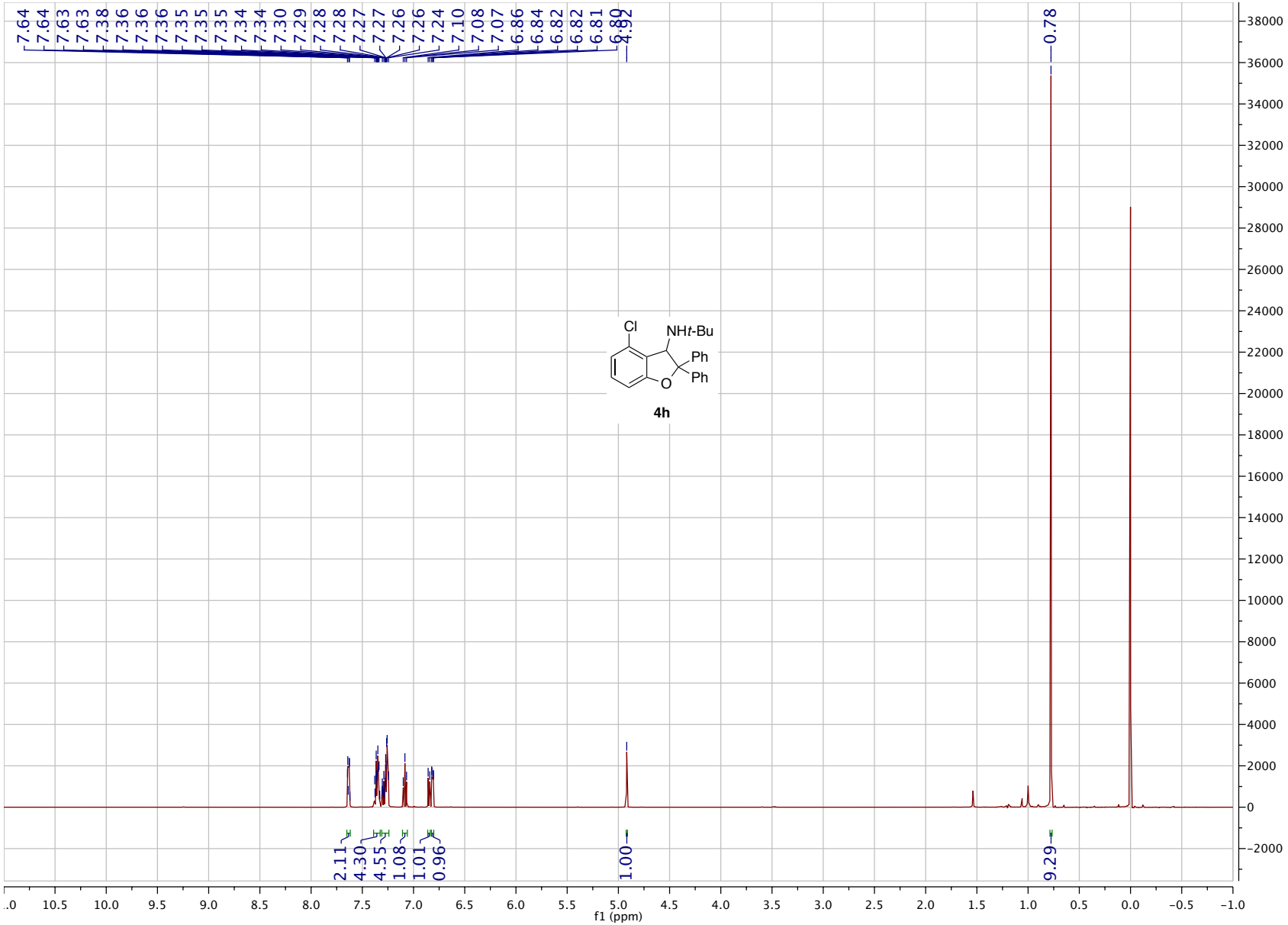
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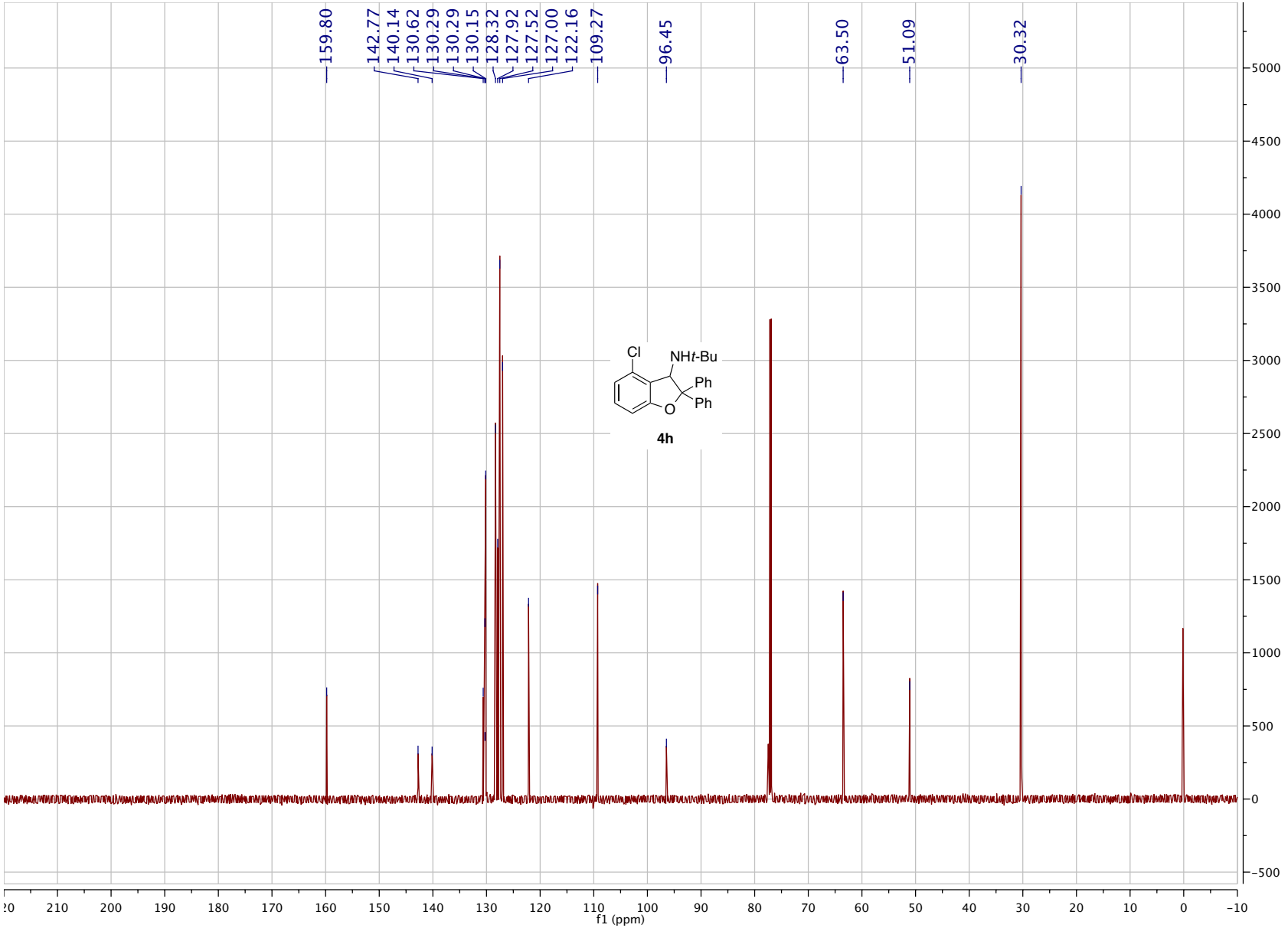
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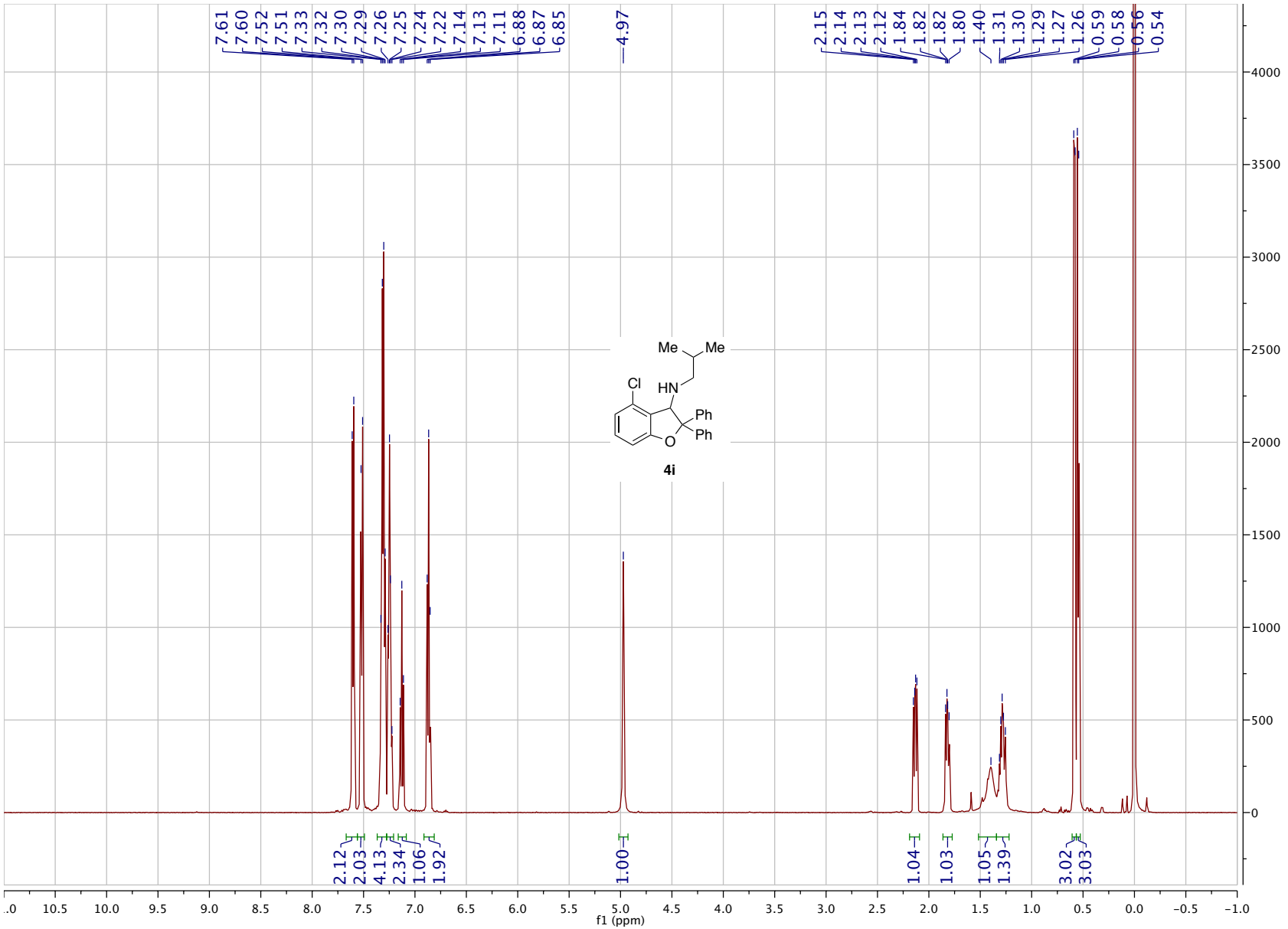
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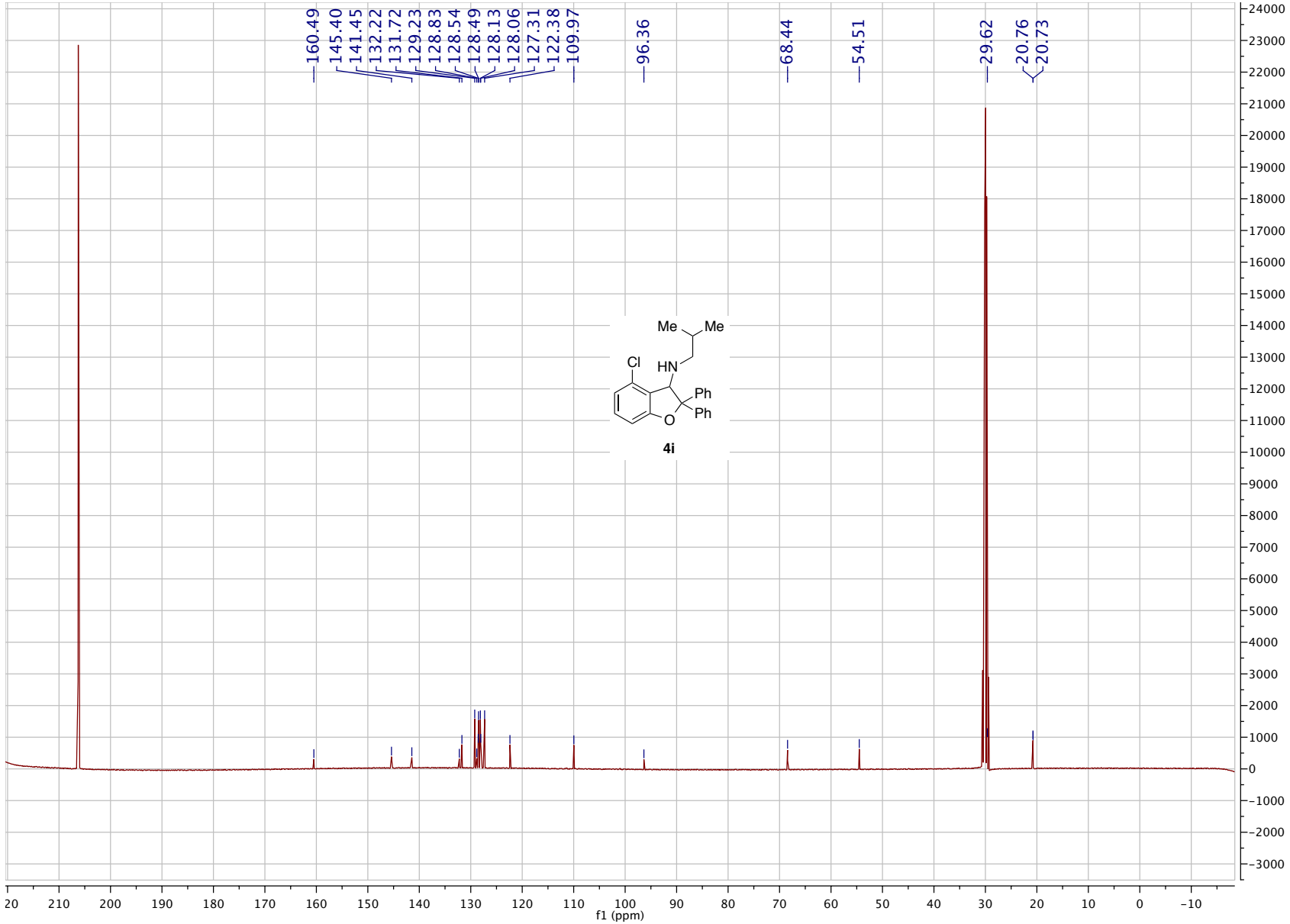
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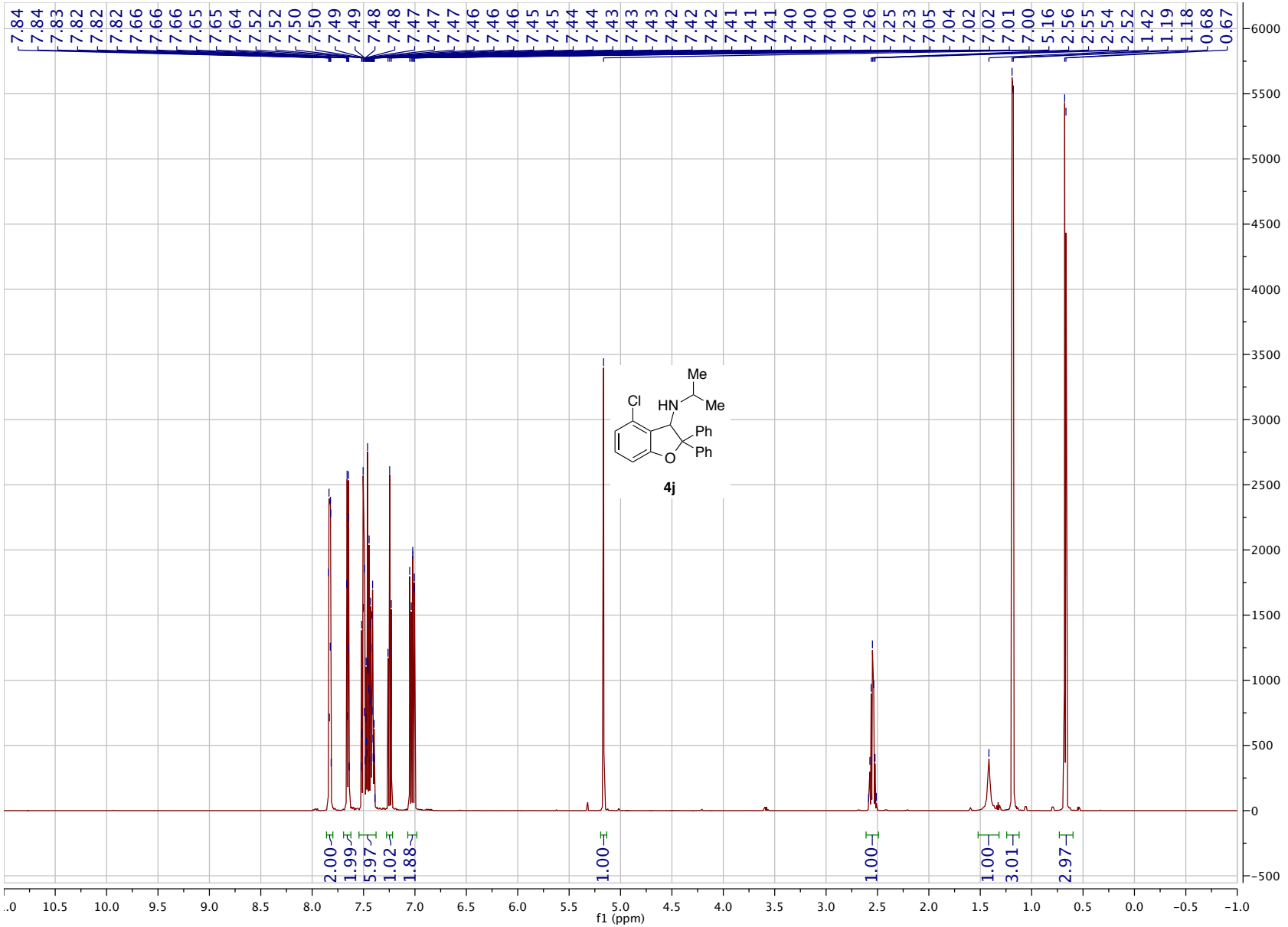
f1 (ppm)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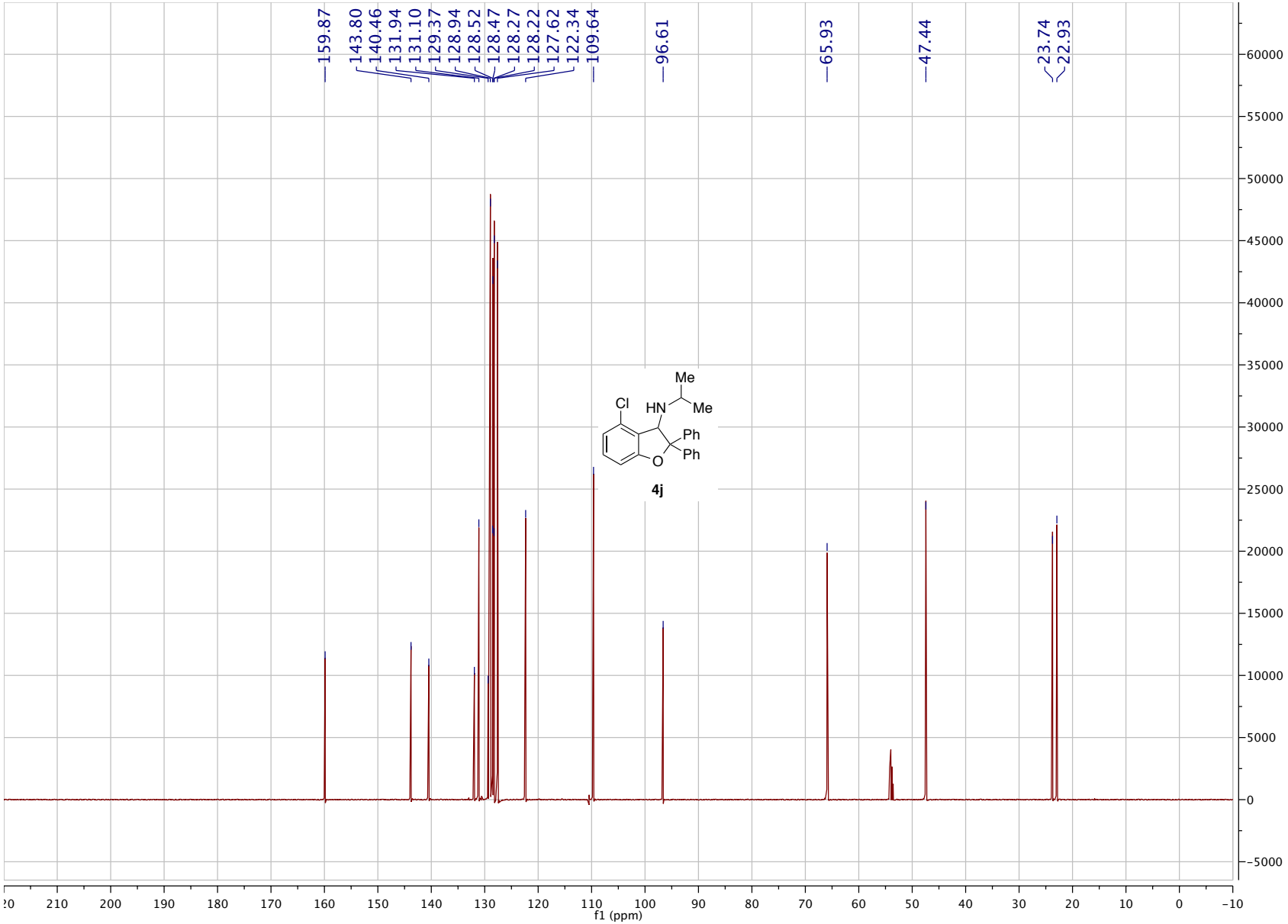
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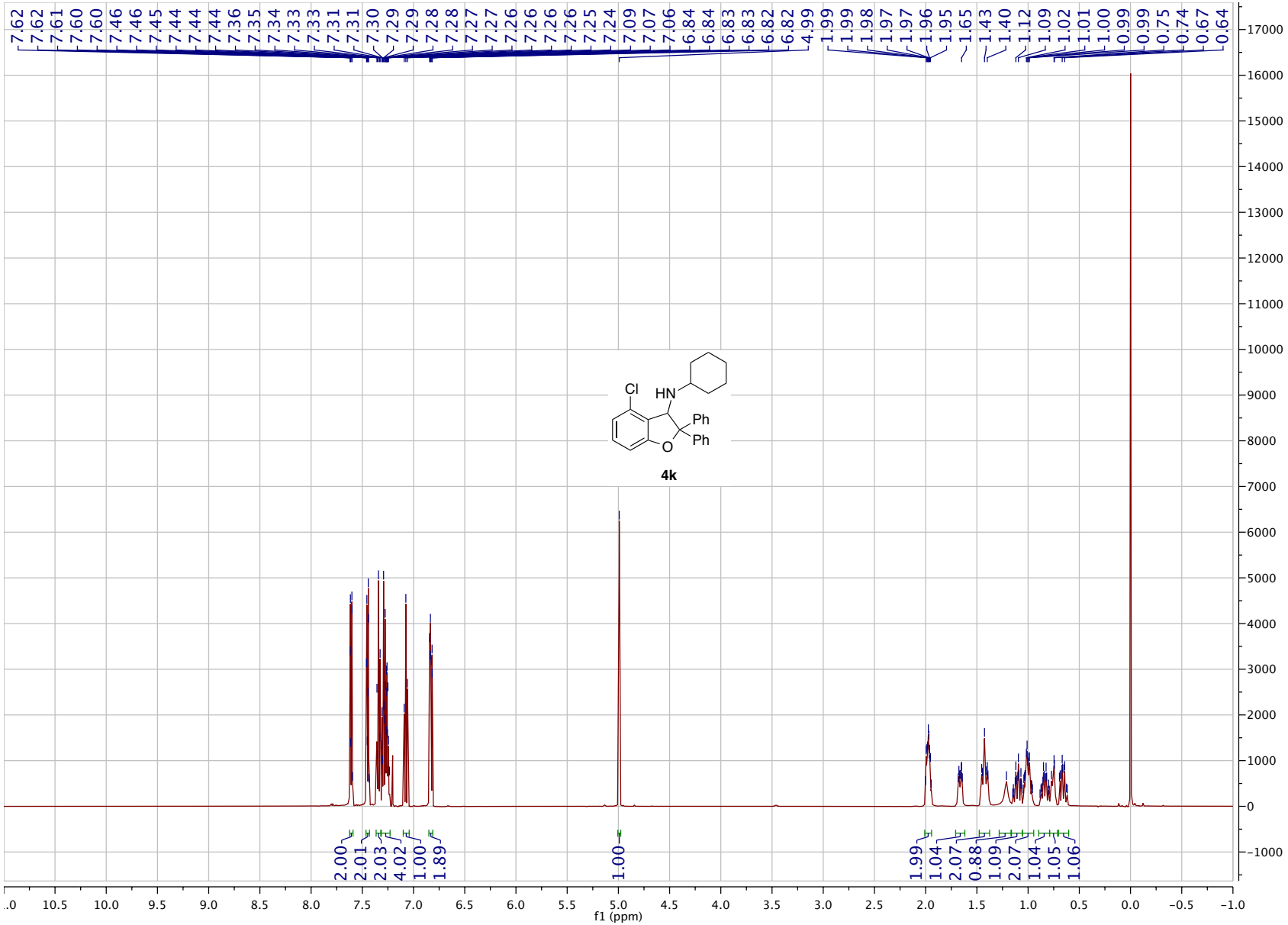
Me
Cl
HN
Me
Ph
Ph
O
4j
f1 (ppm)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Cl
HN
Ph
Ph
O
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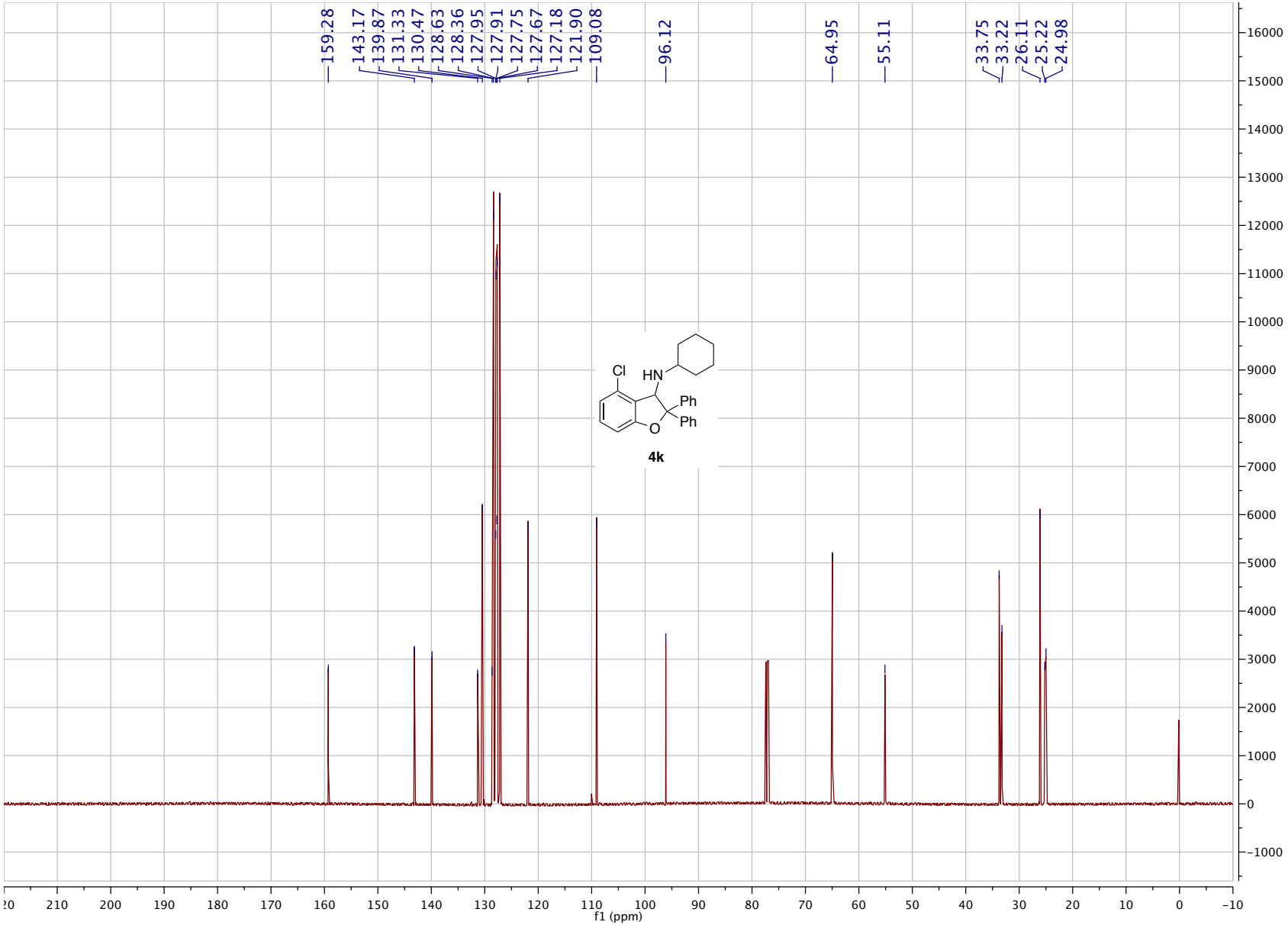
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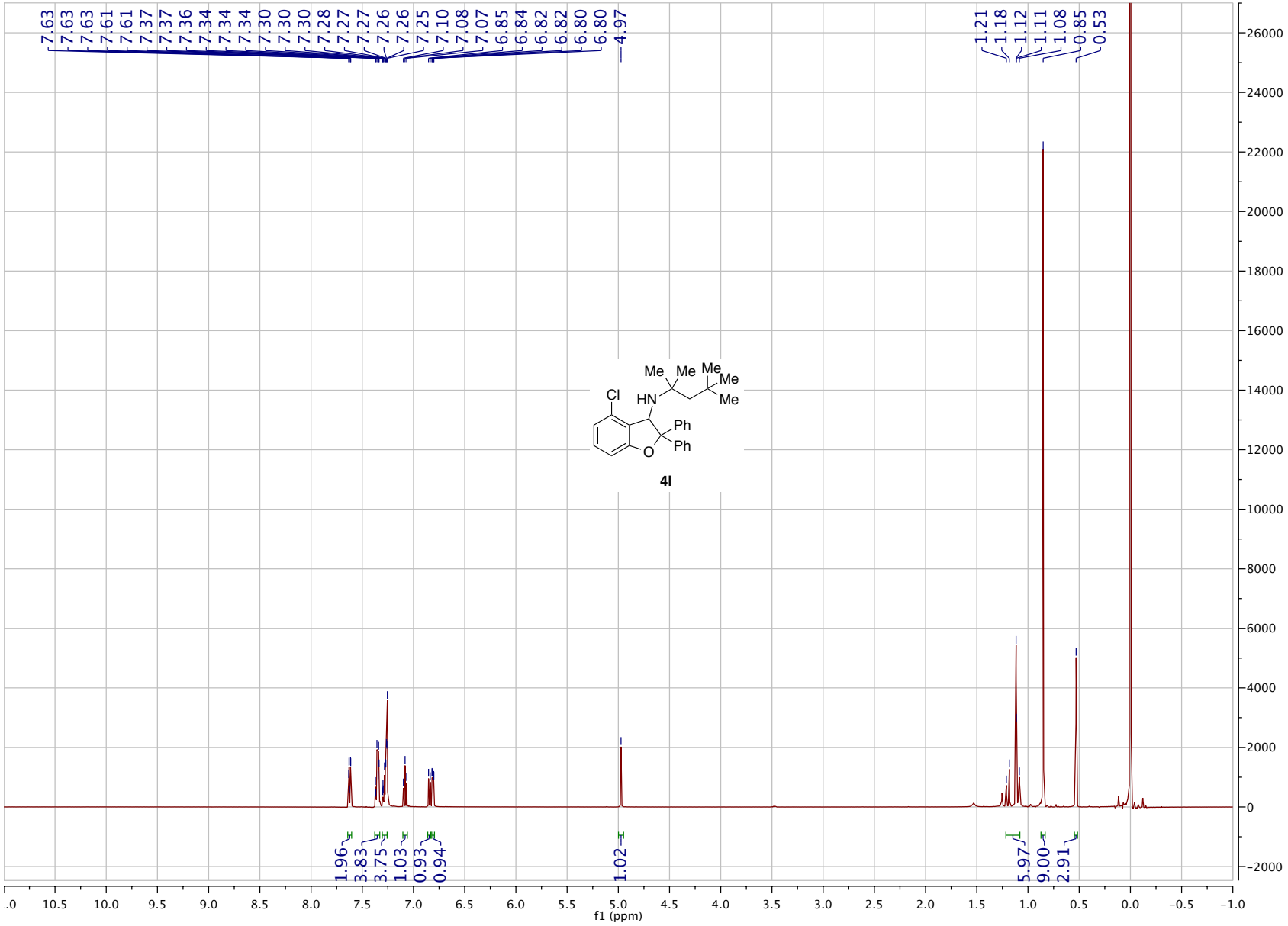
Cl
HN
Ph
Ph
O
4k
f1 (ppm)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6.84
6.82
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6.80
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1.18
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Me
Me
Me
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Cl
HN
Ph
Ph
O
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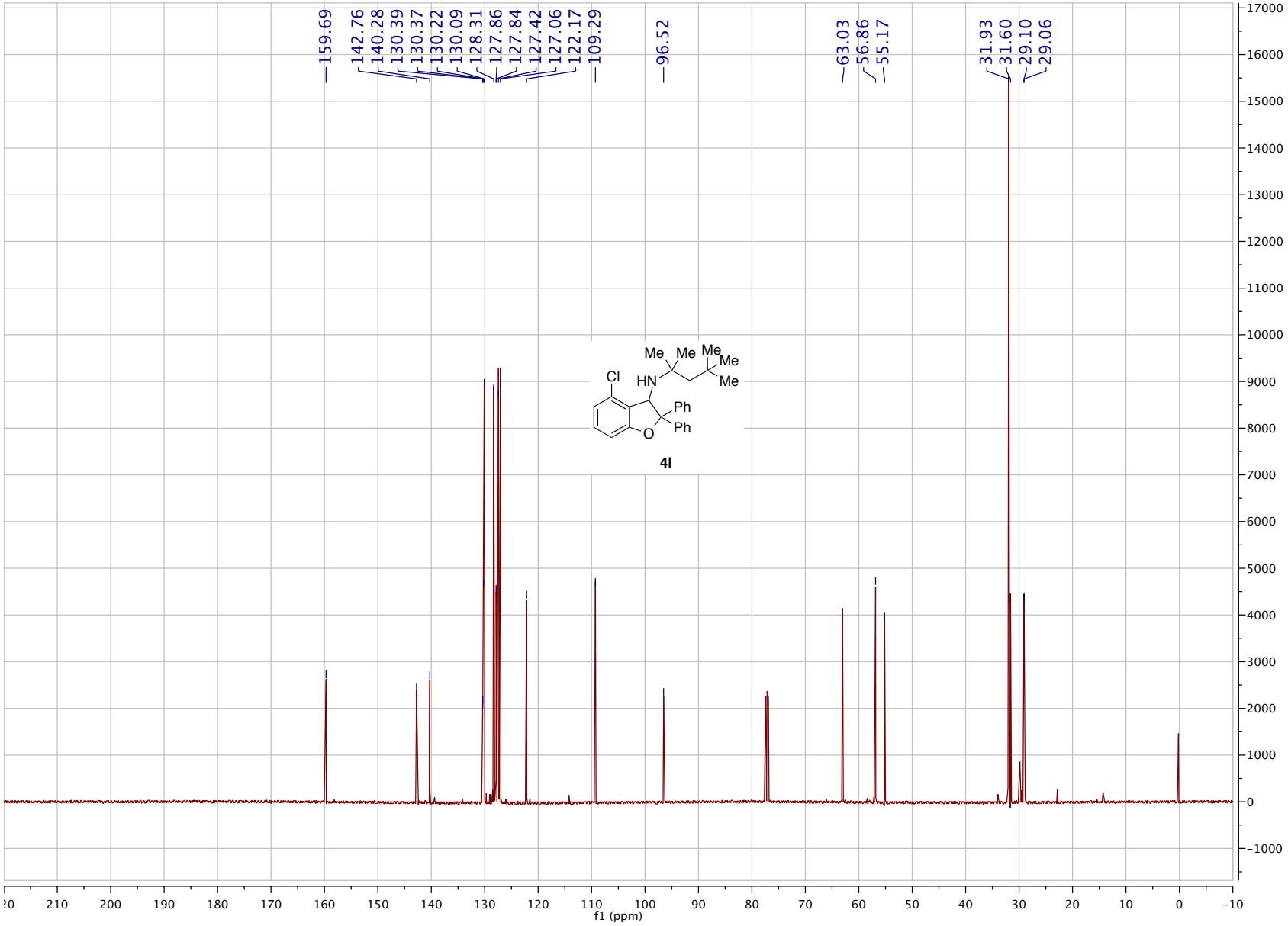
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Ph
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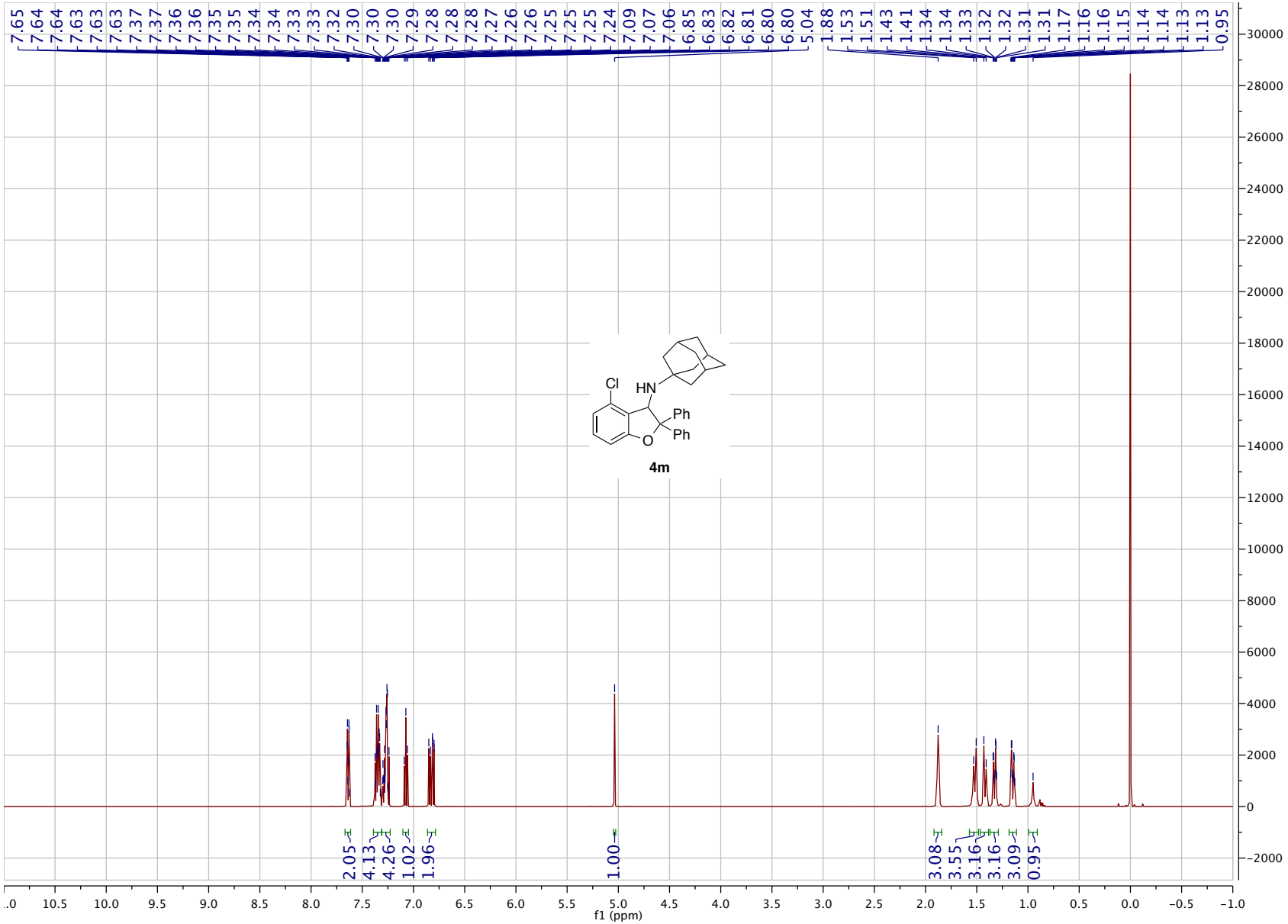
4l
f1 (ppm)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Cl
HN
Ph
Ph
O
4m
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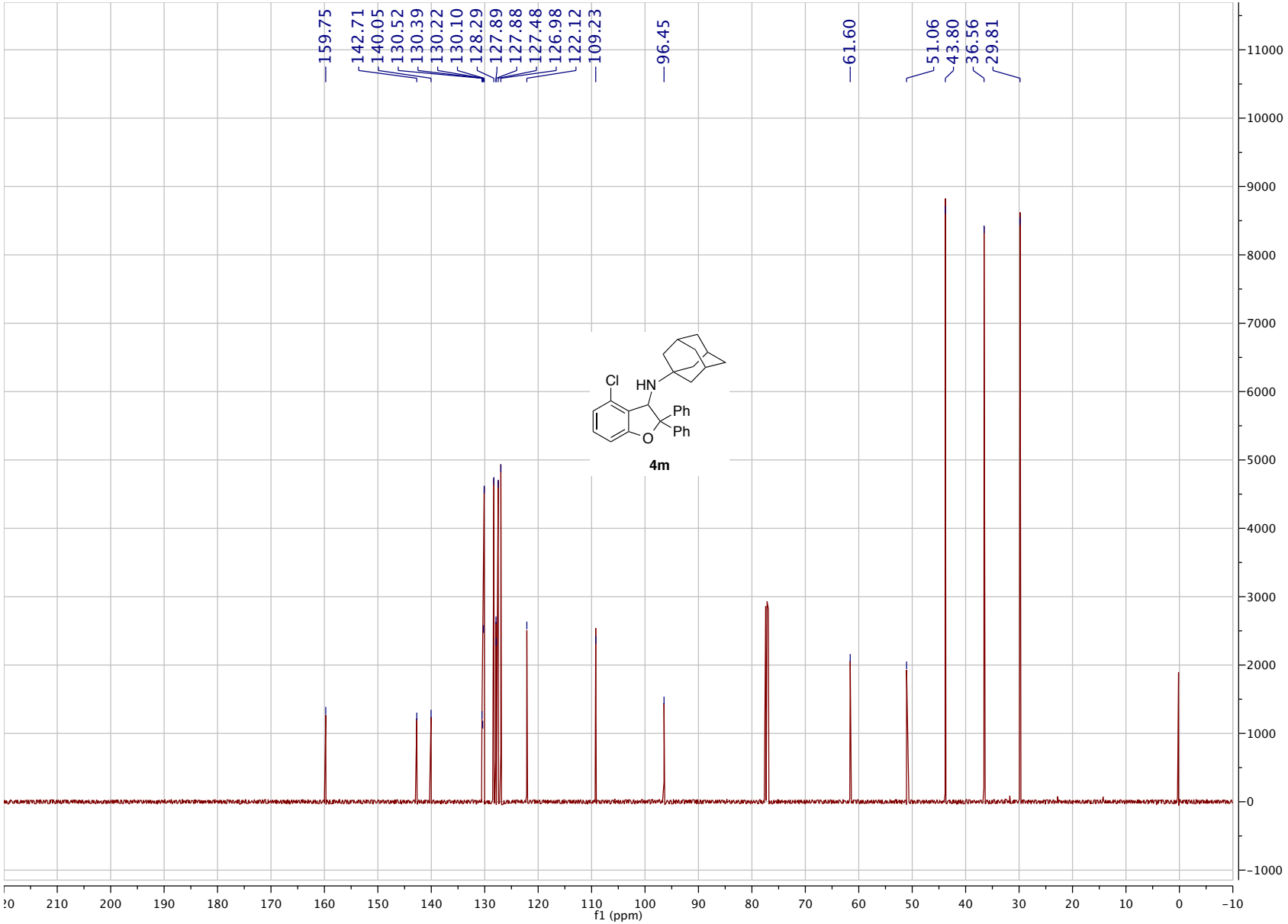
f1 (ppm)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51.06
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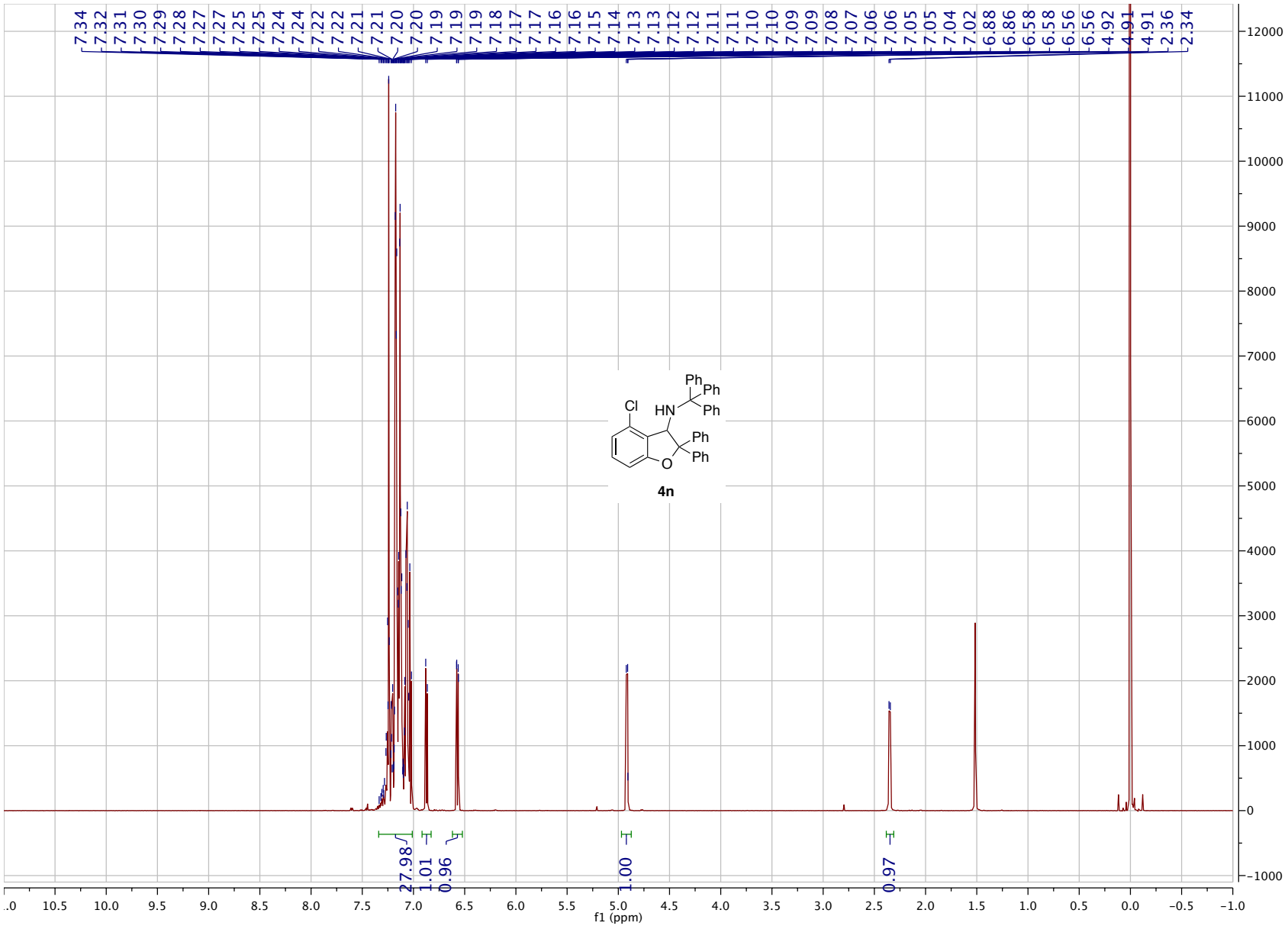
36.56
29.81
Cl
HN
Ph
Ph
O
4m
f1 (ppm)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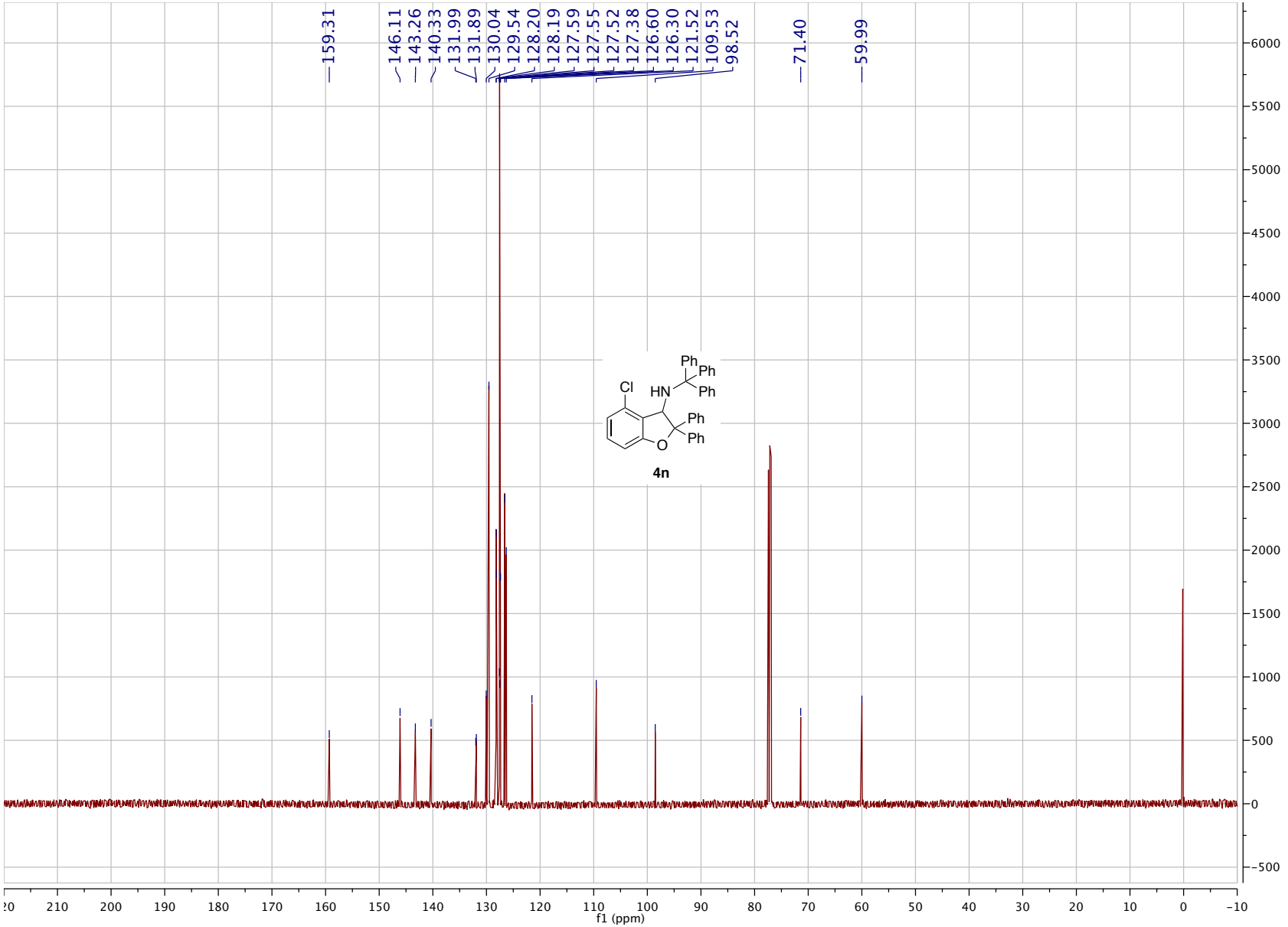
Cl
HN
Ph
Ph
Ph
Ph
Ph
O
4n
27.98
1.01
0.96
1.00
0.97
f1 (ppm)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127.38
126.60
126.30
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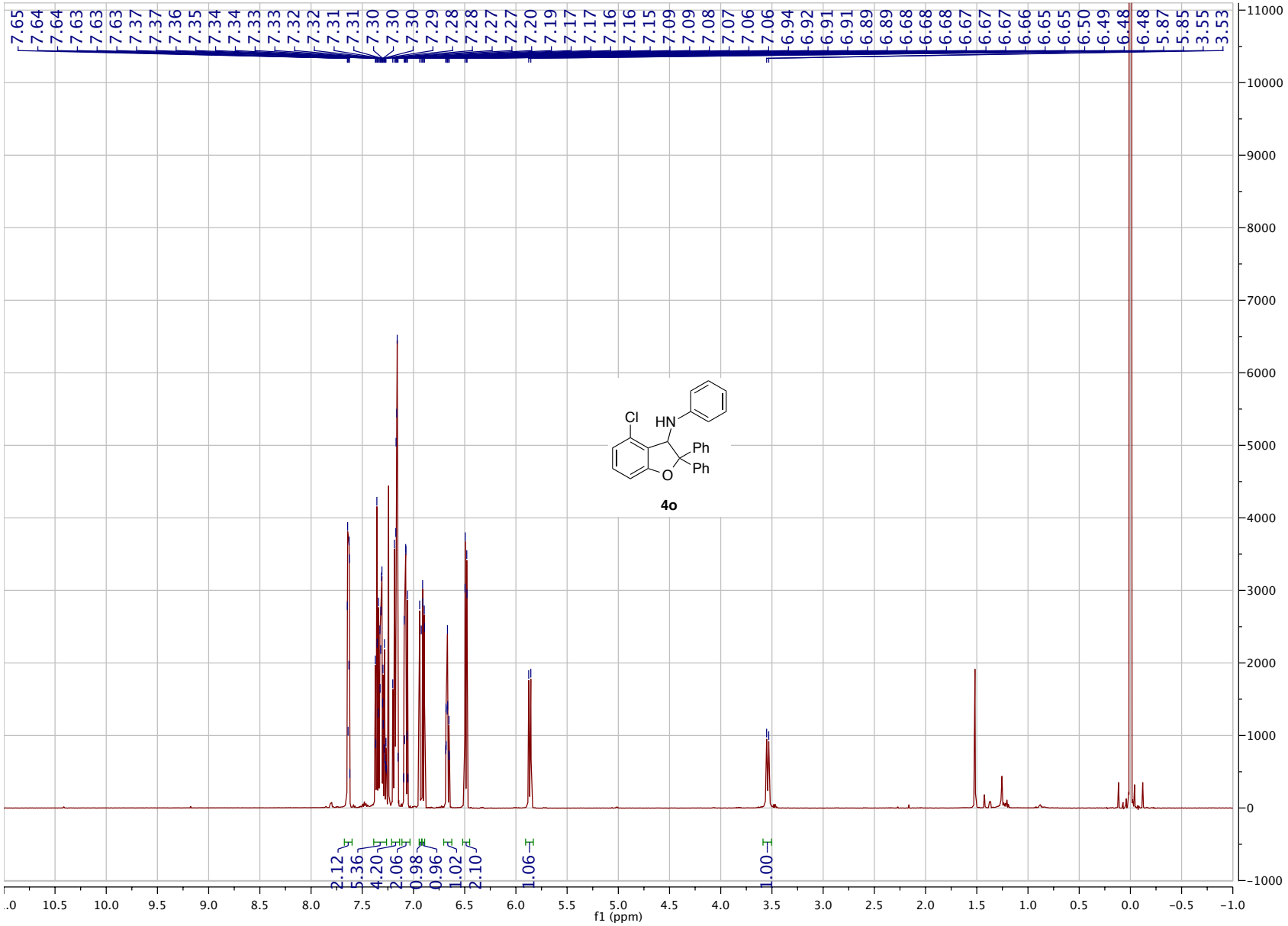
71.40
59.99
Cl
HN
Ph
Ph
Ph
Ph
Ph
O
4n
f1 (ppm)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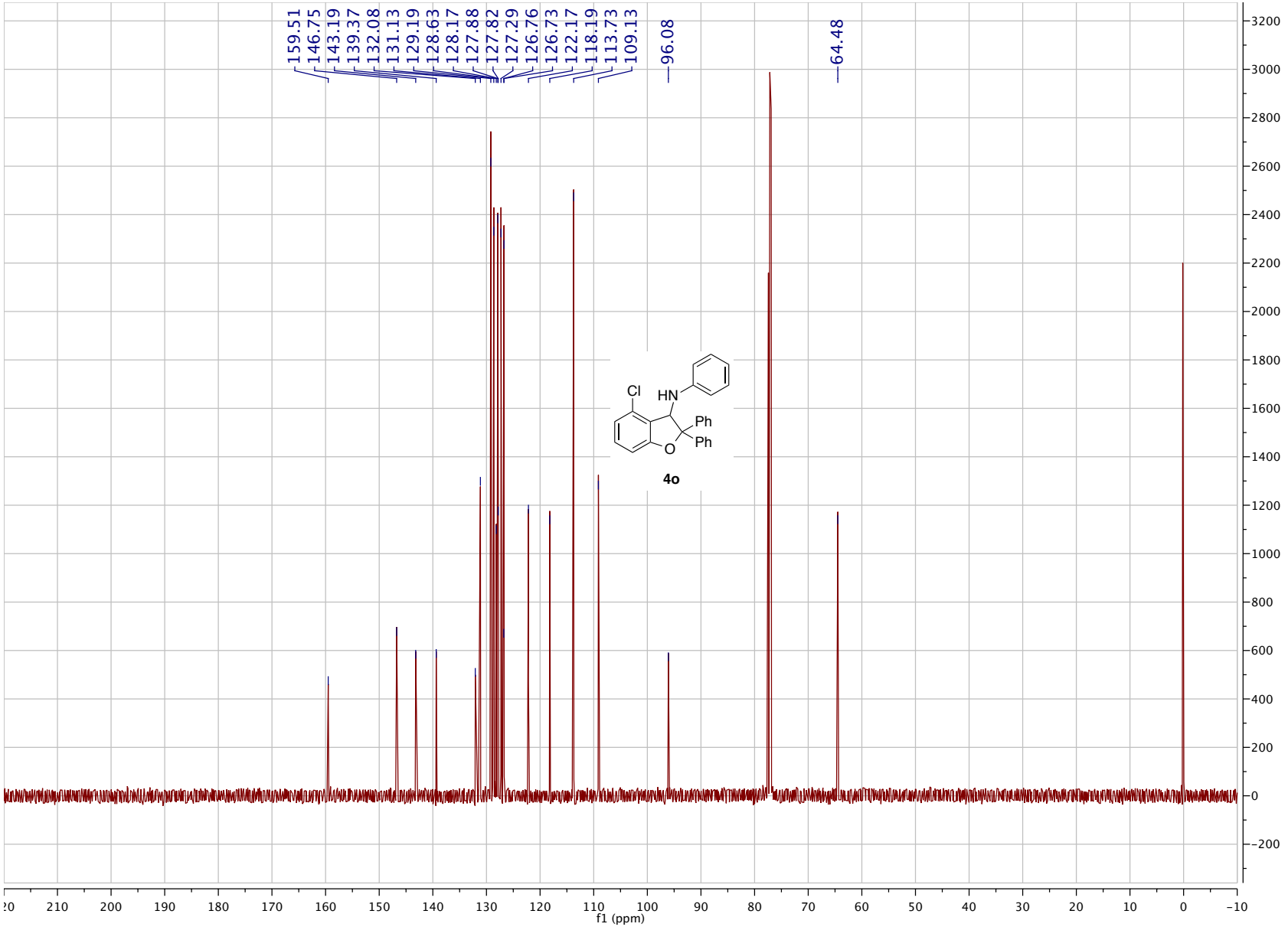
Cl
HN
Ph
Ph
O
4o
2.12 5.36 4.20 2.06 0.98 0.96 1.02 2.10 1.06 1.00
f1 (ppm)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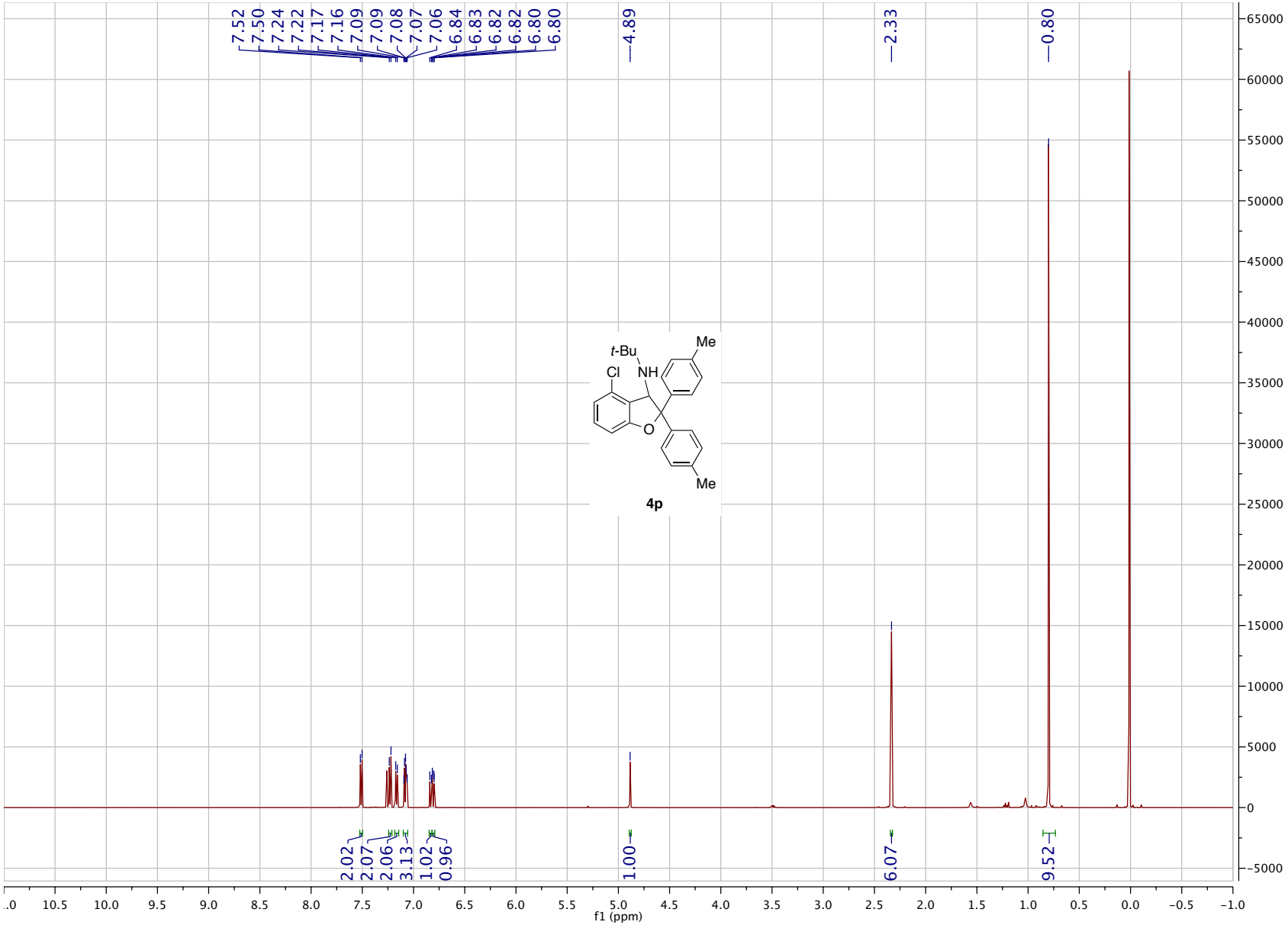
96.08
64.48
Cl
HN
Ph
Ph
O
4o
f1 (ppm)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6.83
6.82
6.82
6.80
6.80
4.89
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0.80
t-Bu
Cl
NH
O
Me
Me
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2.07
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1.00
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9.52
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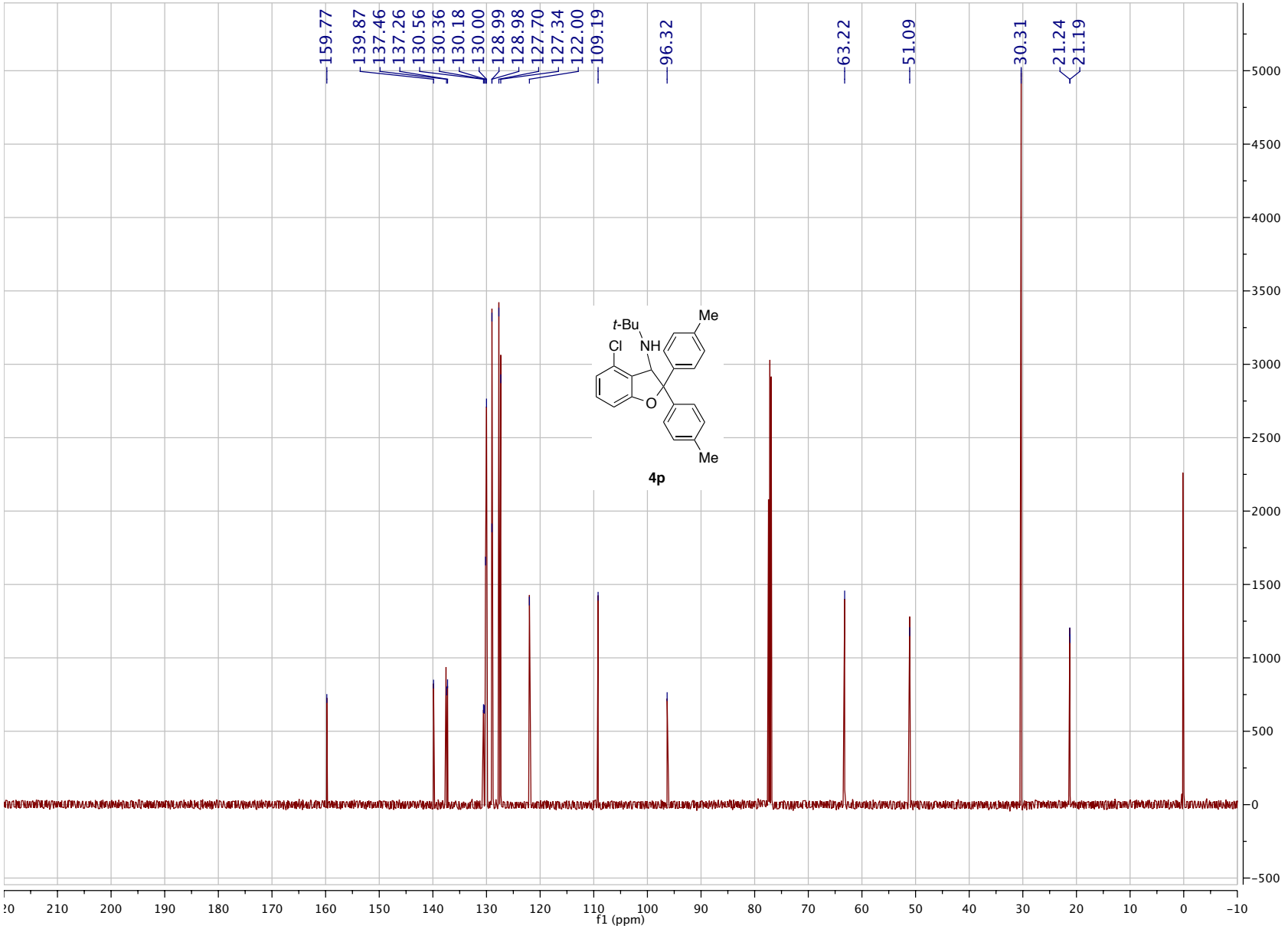
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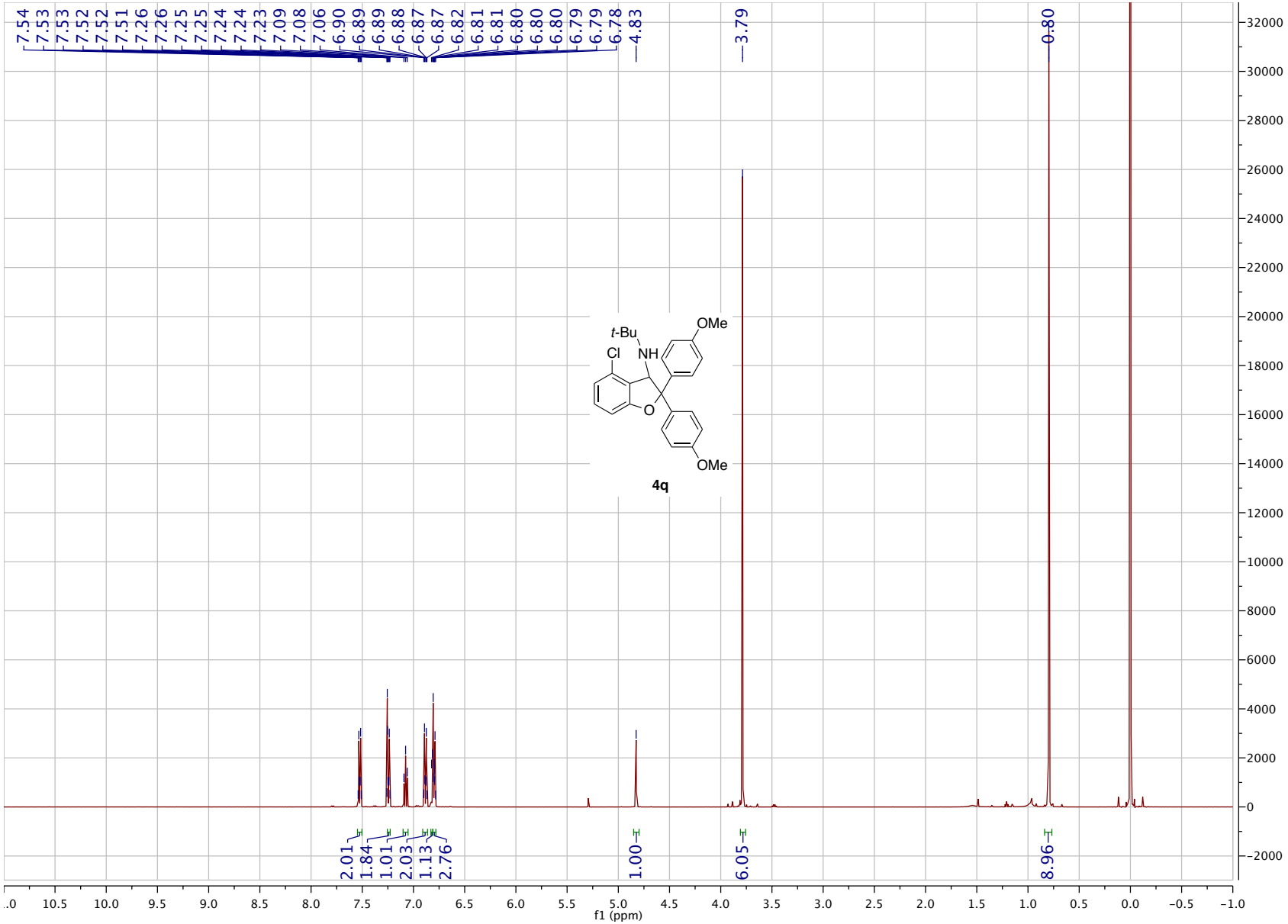
30.31
21.24
21.19
t-Bu
Cl
NH
O
Me
Me
4p
f1 (ppm)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7.08
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6.87
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6.81
6.81
6.80
6.80
6.80
6.79
6.79
6.78
4.83
3.79
0.80
t-Bu
Cl
NH
O
OMe
OMe
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8.96
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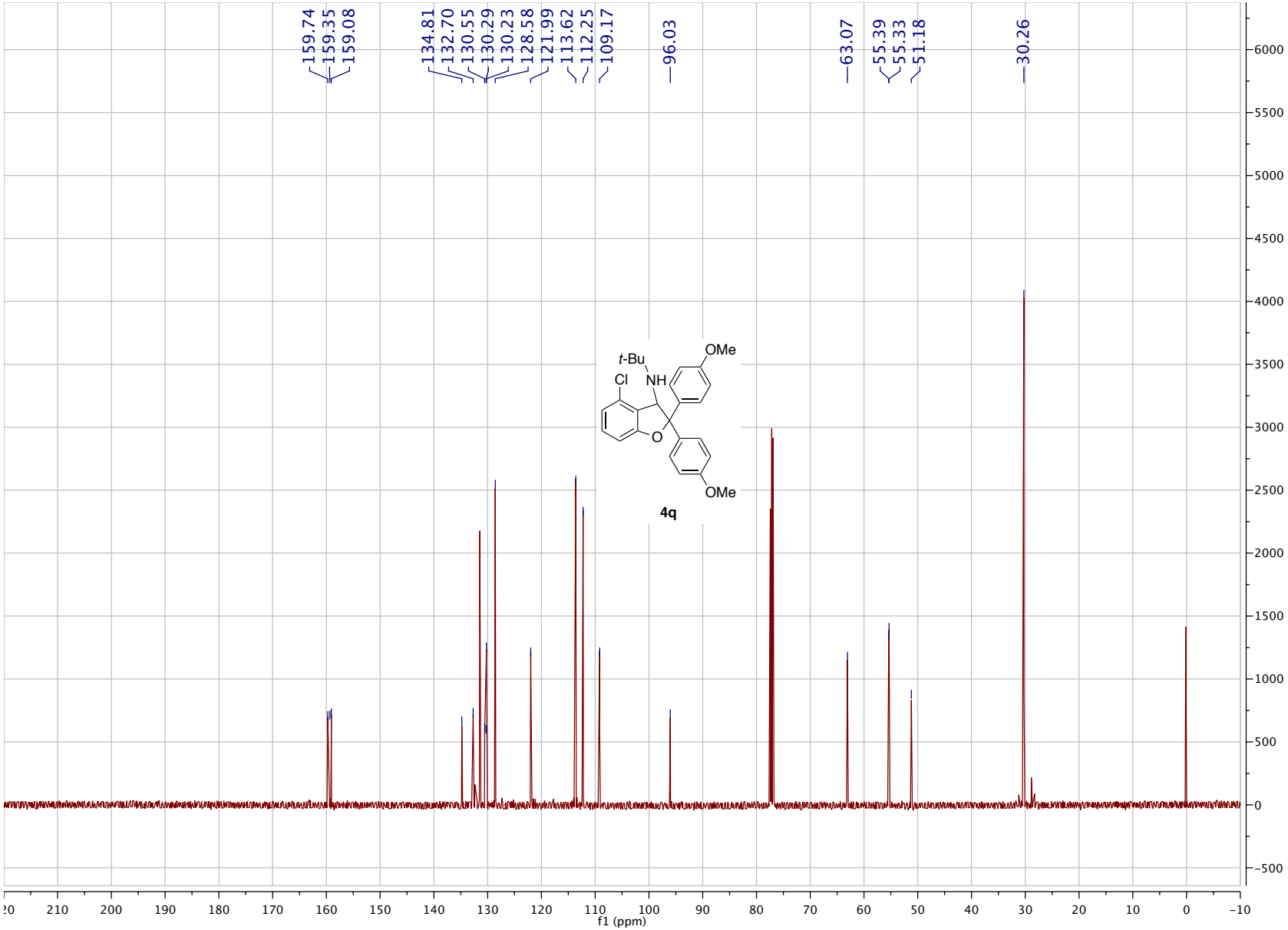
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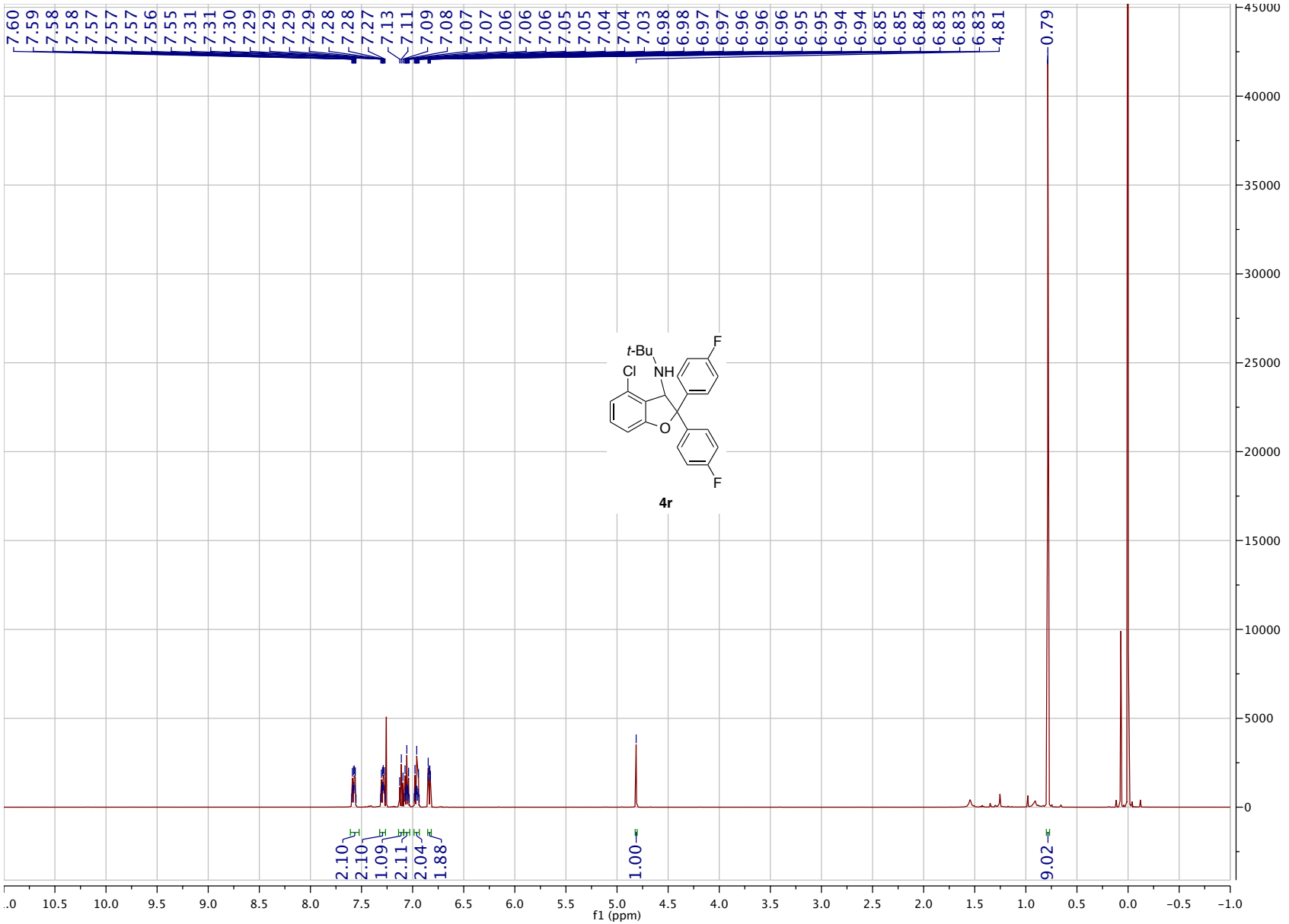
55.33
51.18
30.26
t-Bu
Cl
NH
O
OMe
OMe
4q
f1 (ppm)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NH
O
F
F
4r
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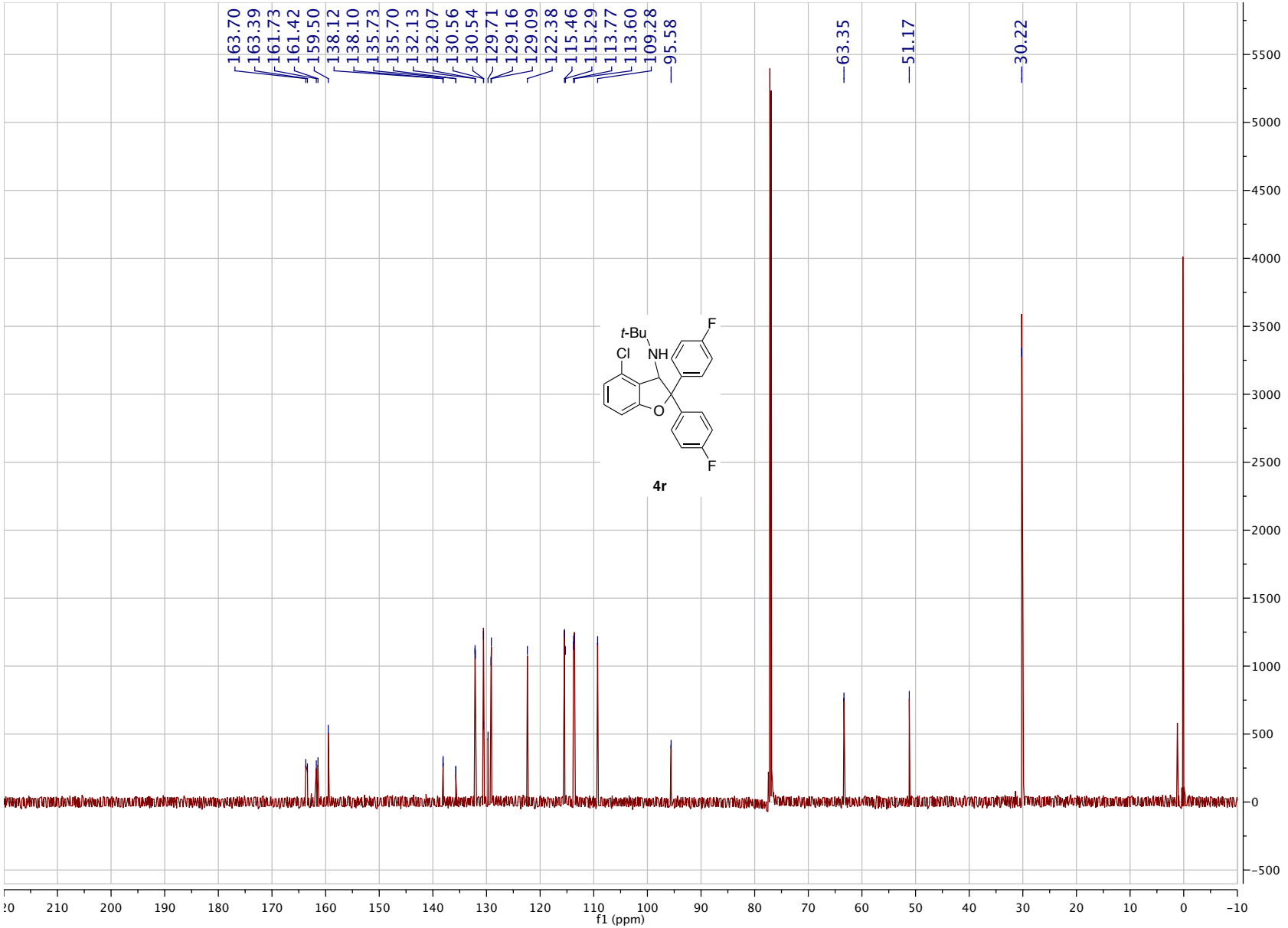
f1 (ppm)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Cl
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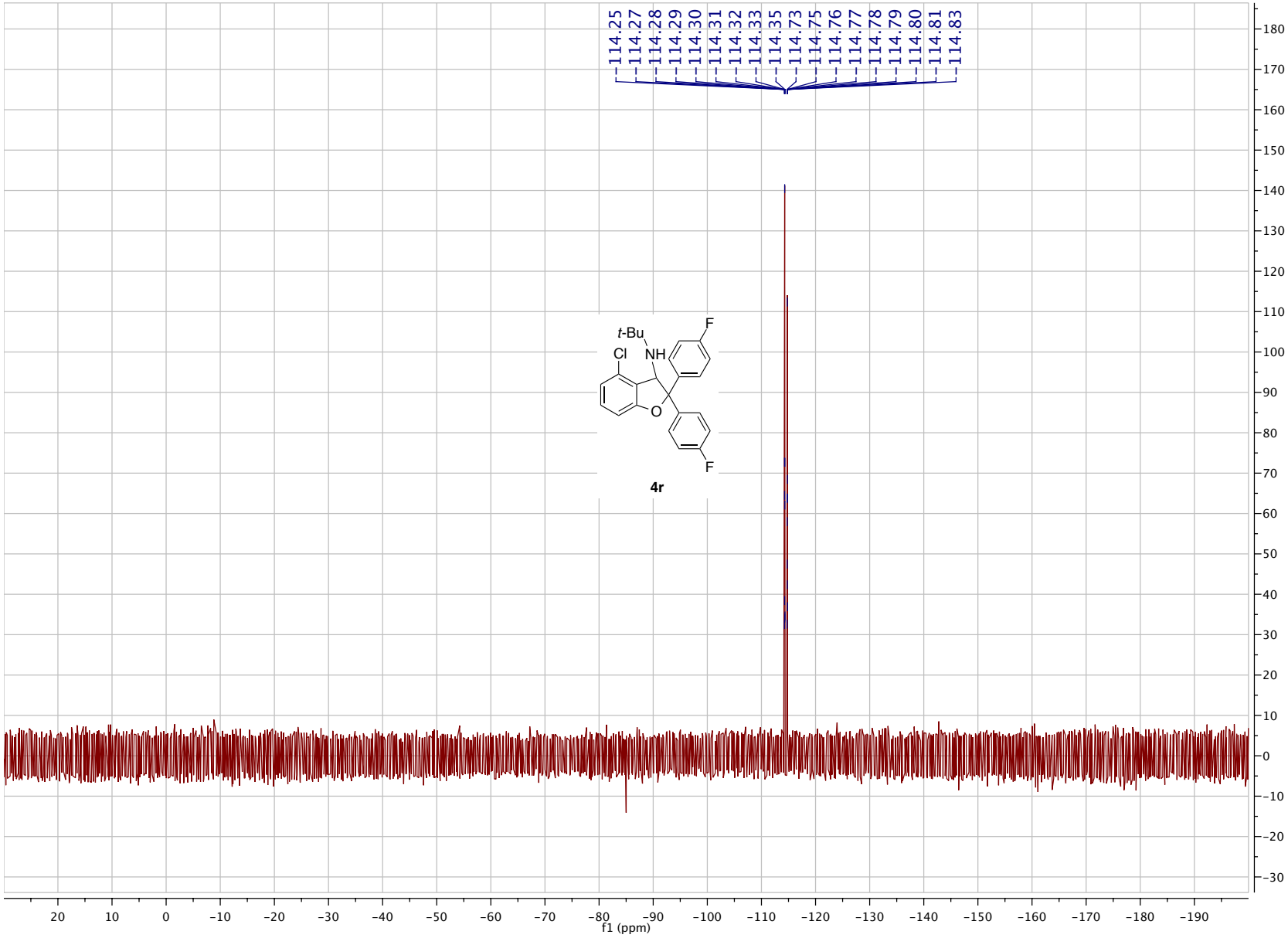
O
F
F
4r
f1 (ppm)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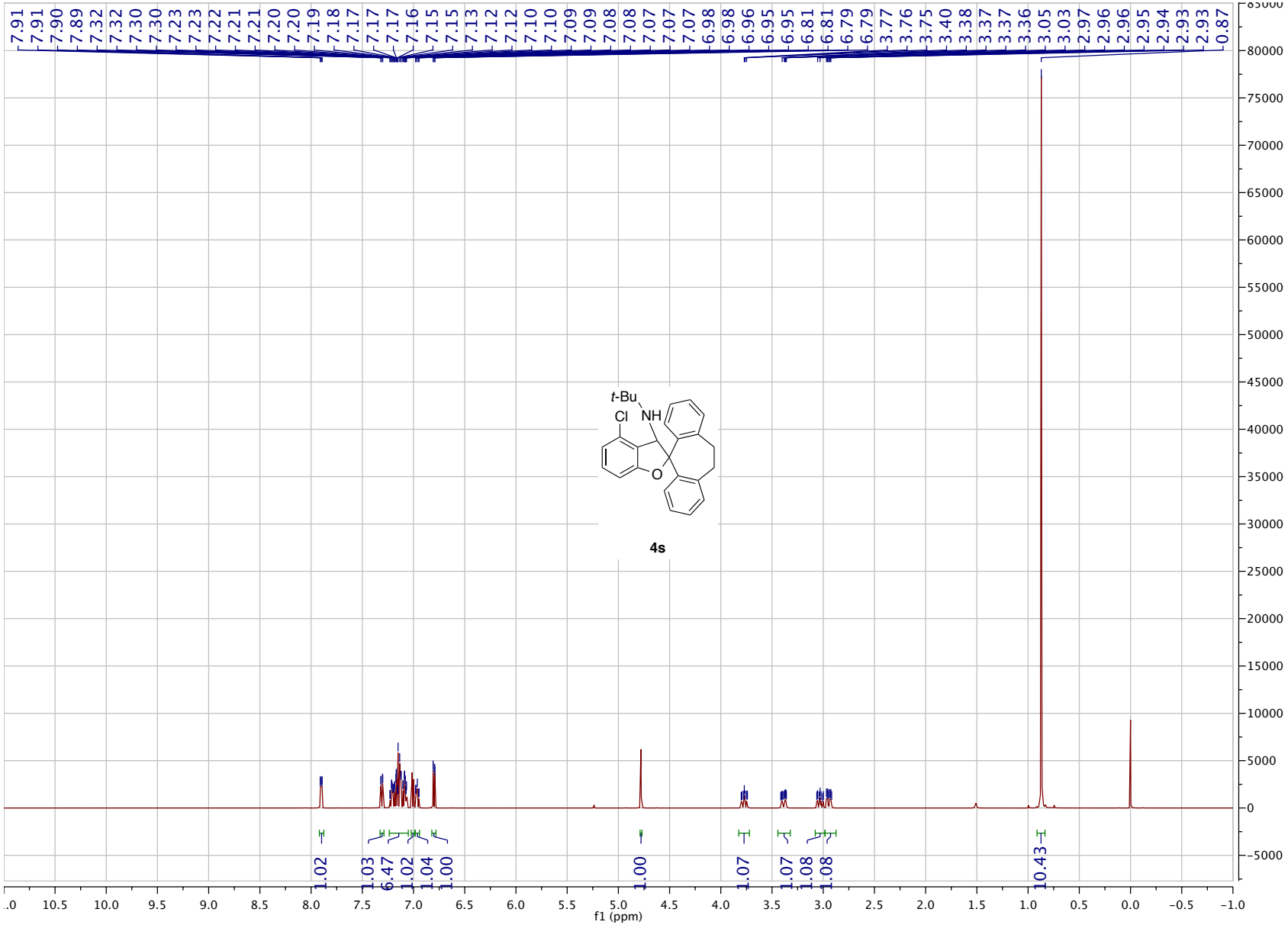
F
4r
f1 (ppm)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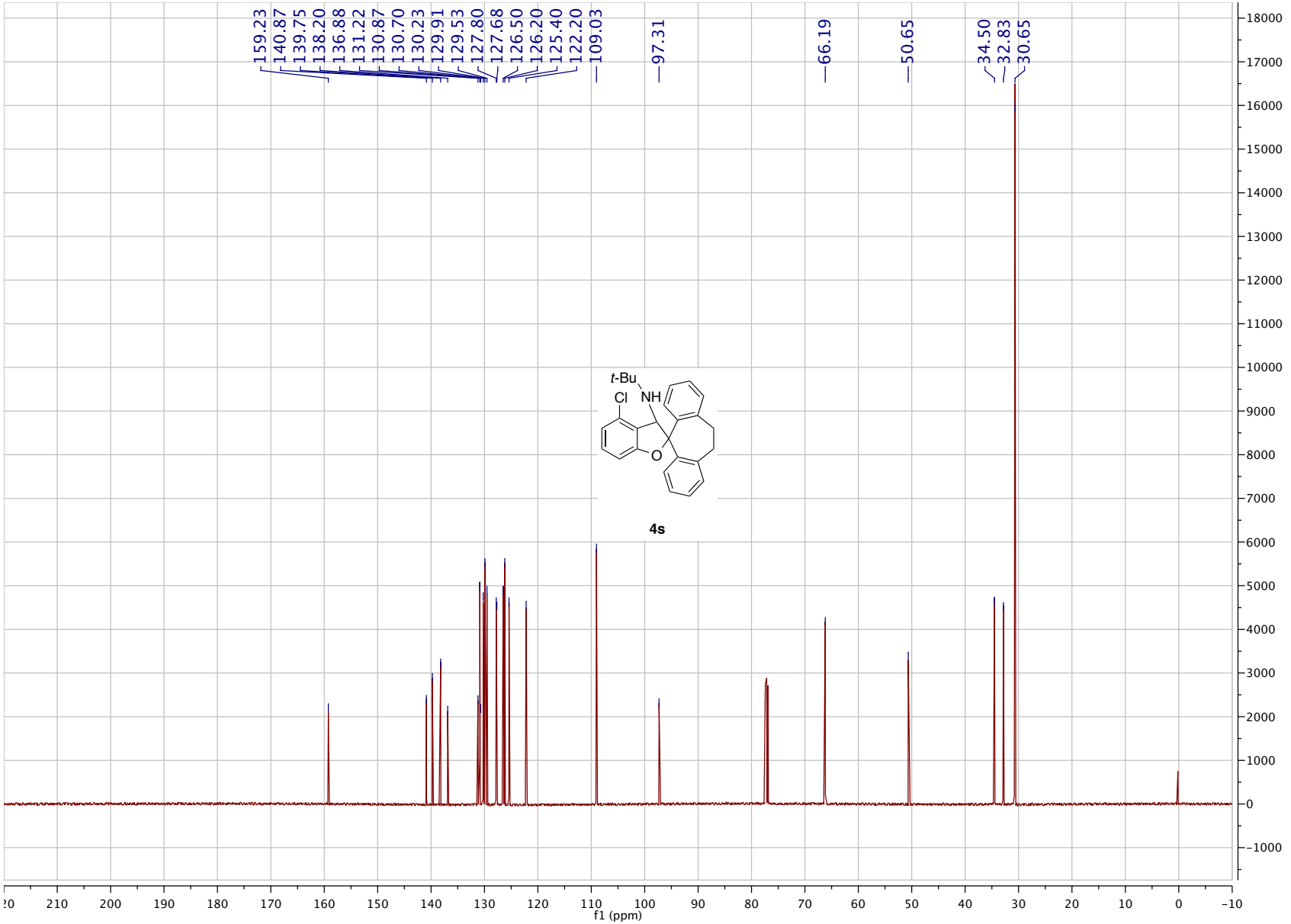
t-Bu
Cl
NH
O
4s
1.02 1.03 6.47 1.02 1.04 1.00 1.00 1.07 1.07 1.08 1.08 10.43
f1 (ppm)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Cl
NH
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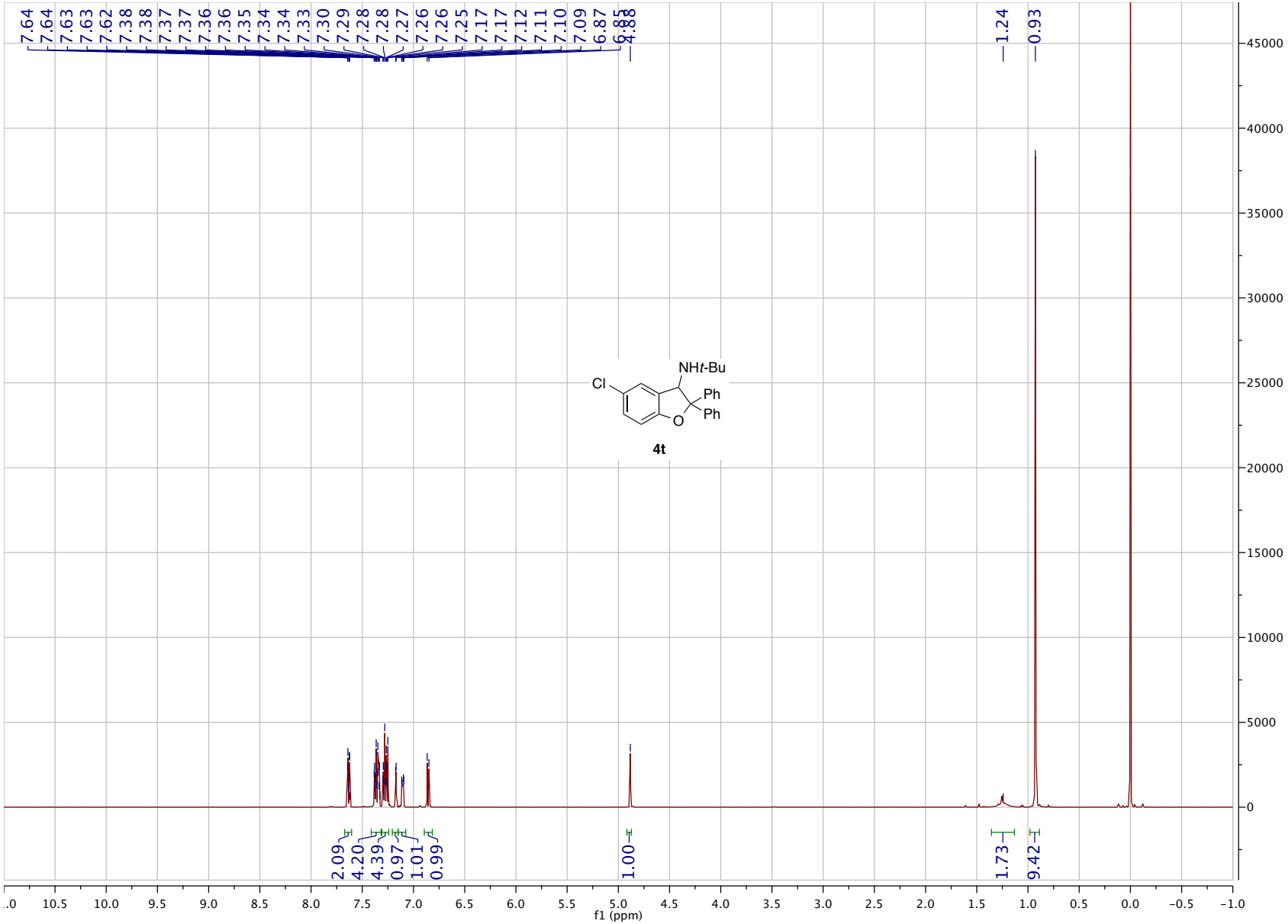
4s
f1 (ppm)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1.24
0.93
NHt-Bu
Cl
Ph
Ph
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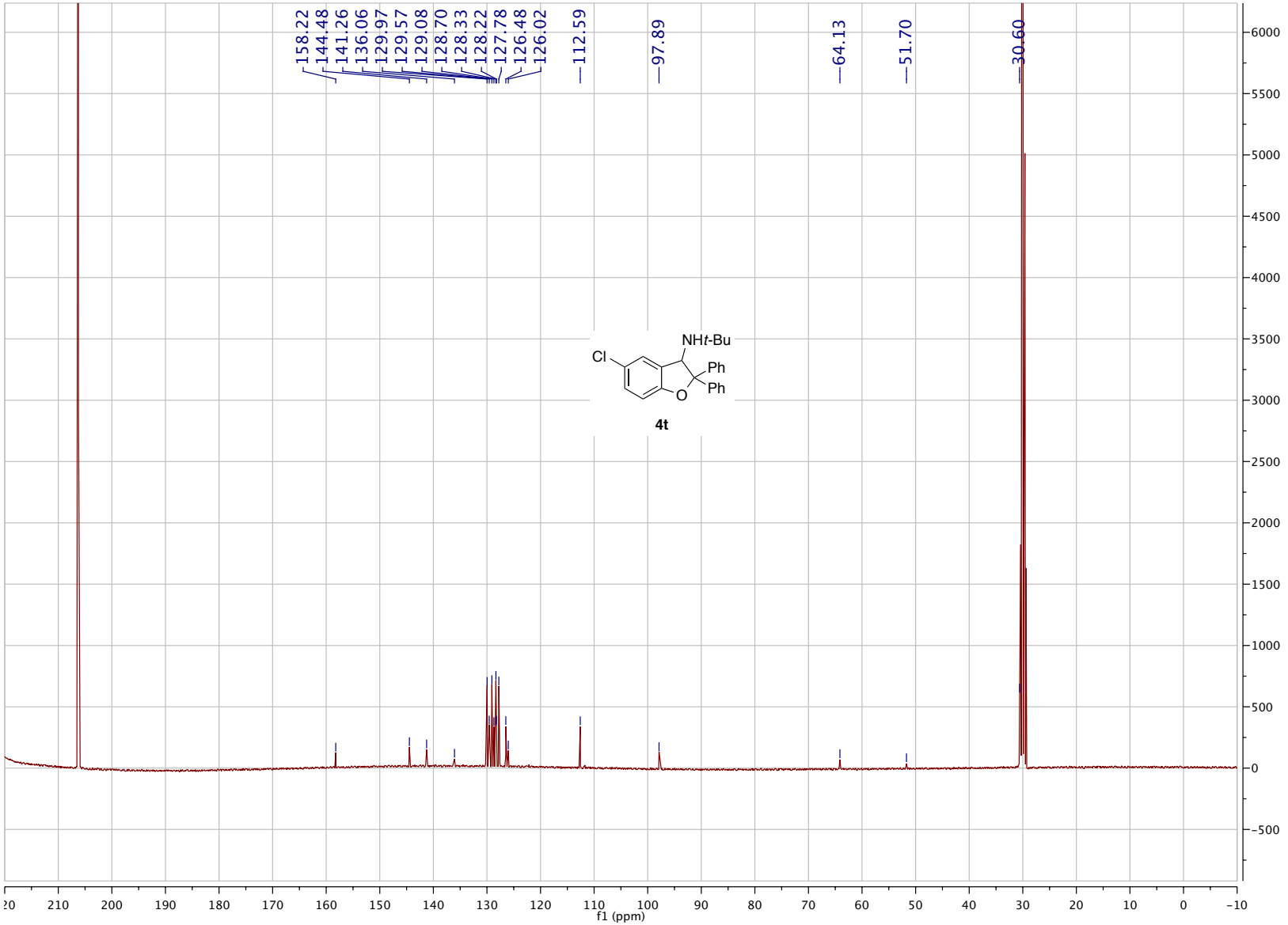
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64.13
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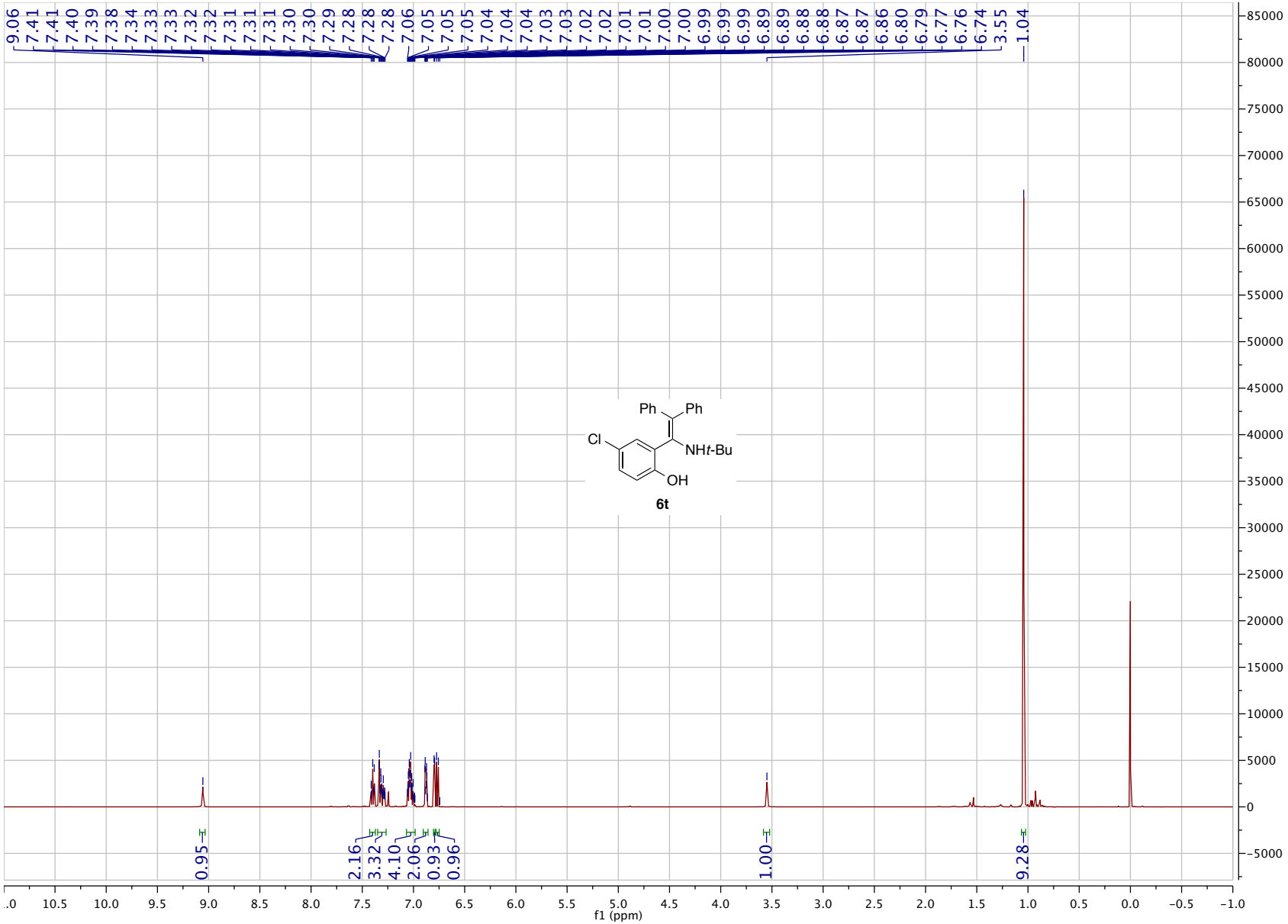
30.60
NHt-Bu
Cl
Ph
Ph
O
4t
f1 (ppm)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Ph
Cl
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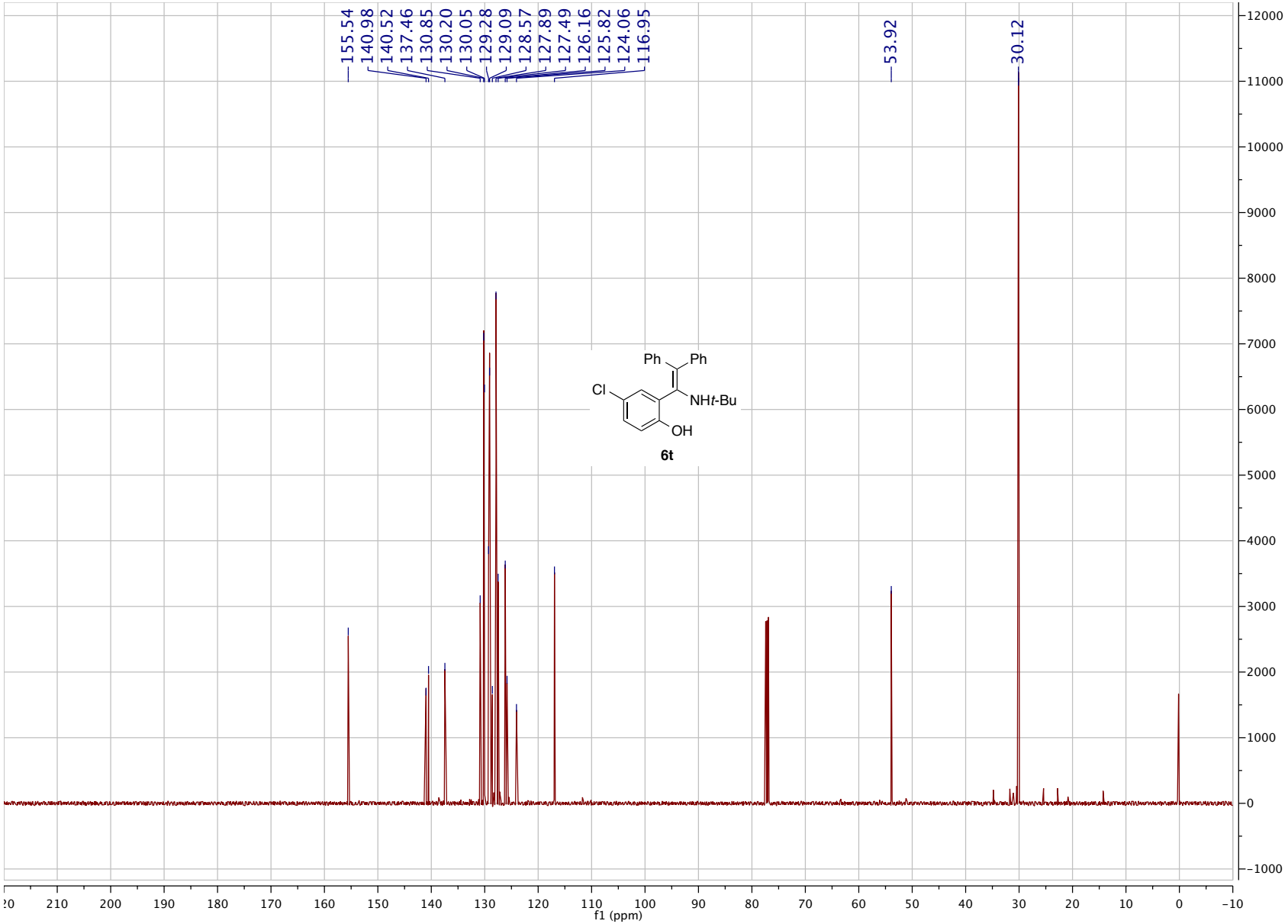
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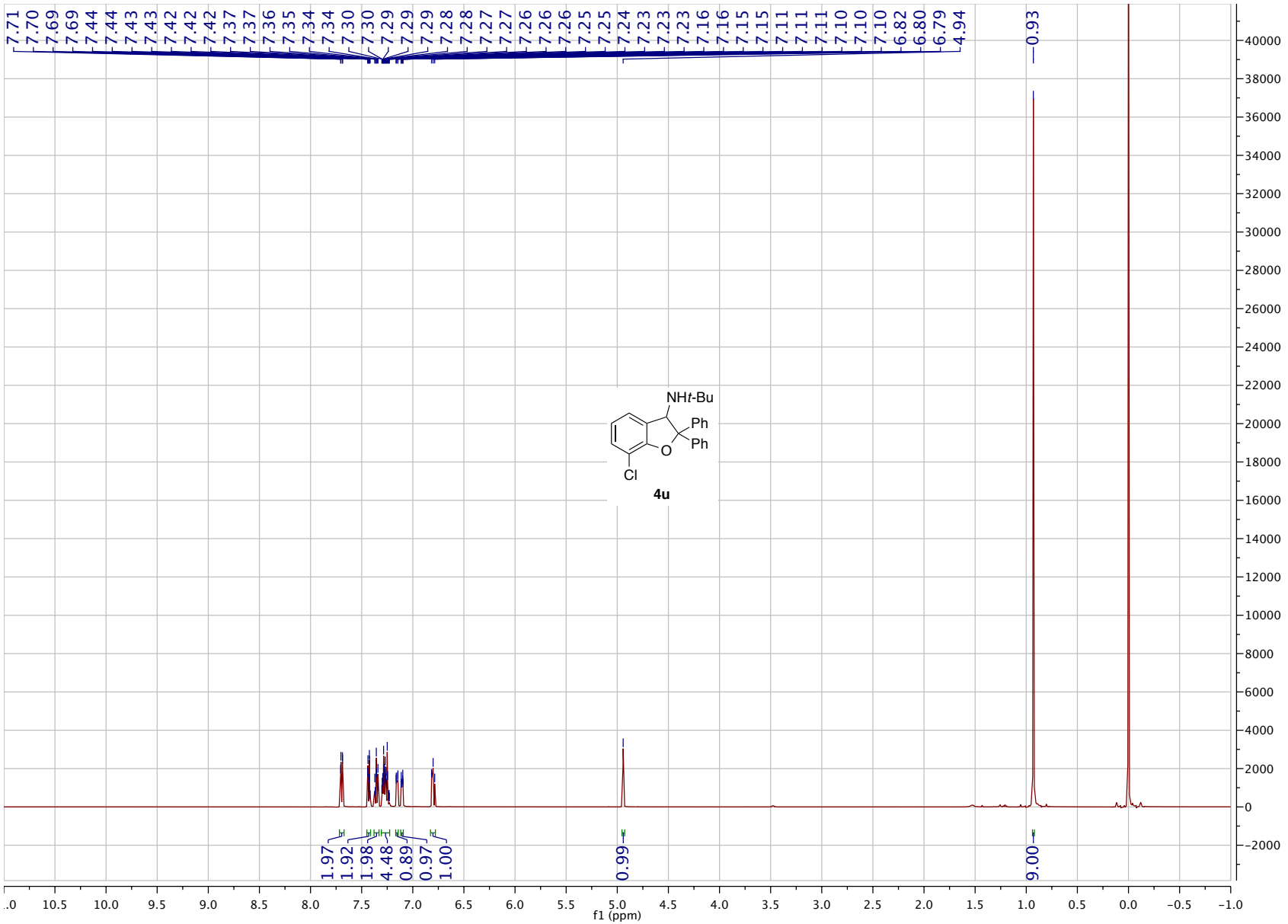
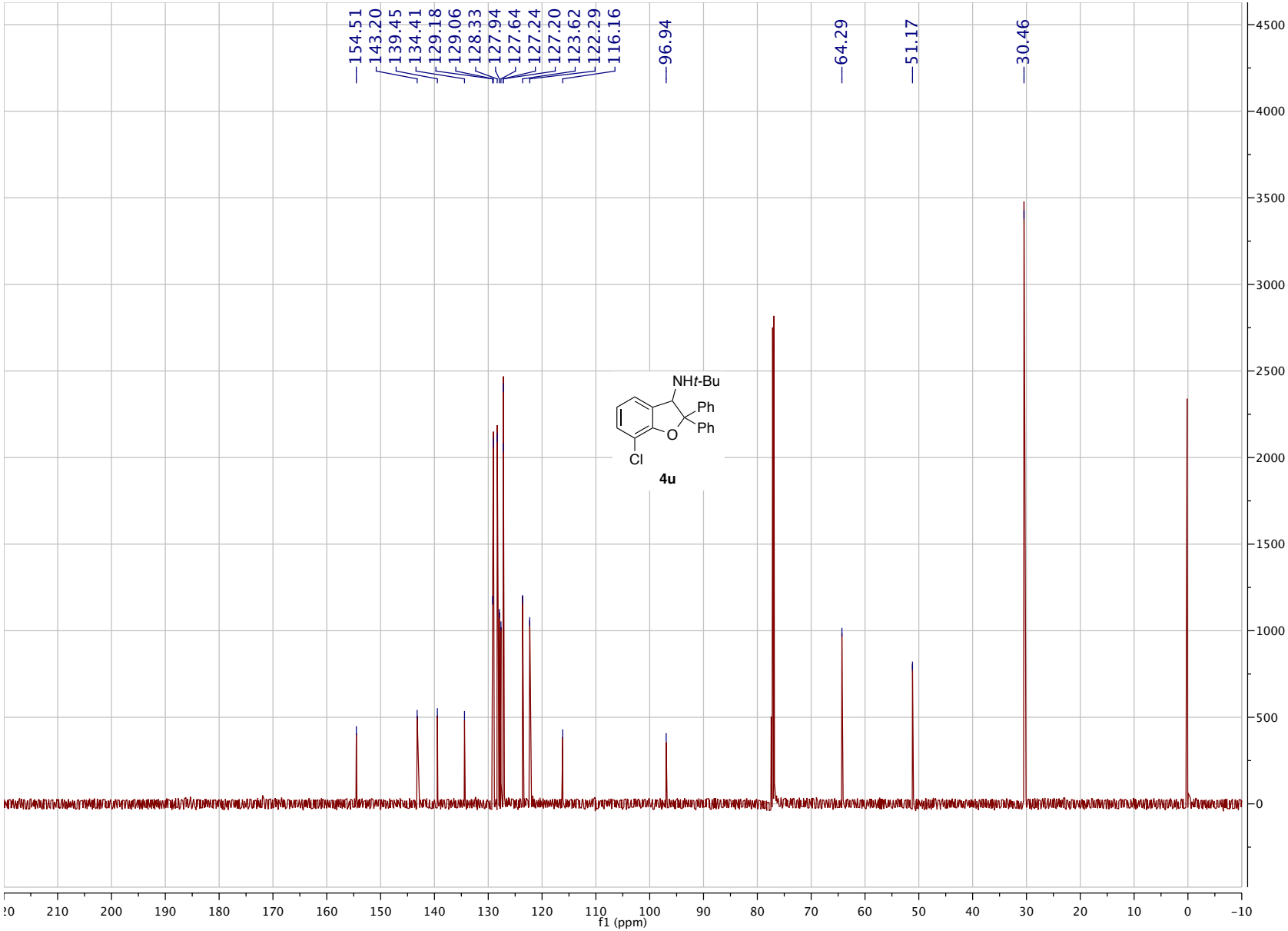
125.82
124.06
116.95
53.92
30.12
Ph
Ph
Cl
NHt-Bu
OH
6t
f1 (ppm)


7.71 7.70 7.69 7.69 7.44 7.44 7.43 7.43 7.42 7.42 7.42 7.37 7.37 7.36 7.35 7.34 7.34 7.30 7.30 7.29 7.29 7.29 7.28 7.28 7.27 7.27 7.26 7.26 7.25 7.25 7.24 7.23 7.23 7.16 7.16 7.15 7.15 7.11 7.11 7.10 7.10 7.10 6.82 6.80 6.79 4.94
0.93
NHt-Bu
Ph
Ph
O
Cl
4u
1.97 1.92 1.98 4.48 0.89 0.97 1.00
0.99
9.00
f1 (ppm)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143.20
139.45
134.41
129.18
129.06
128.33
127.94
127.64
127.24
127.20
123.62
122.29
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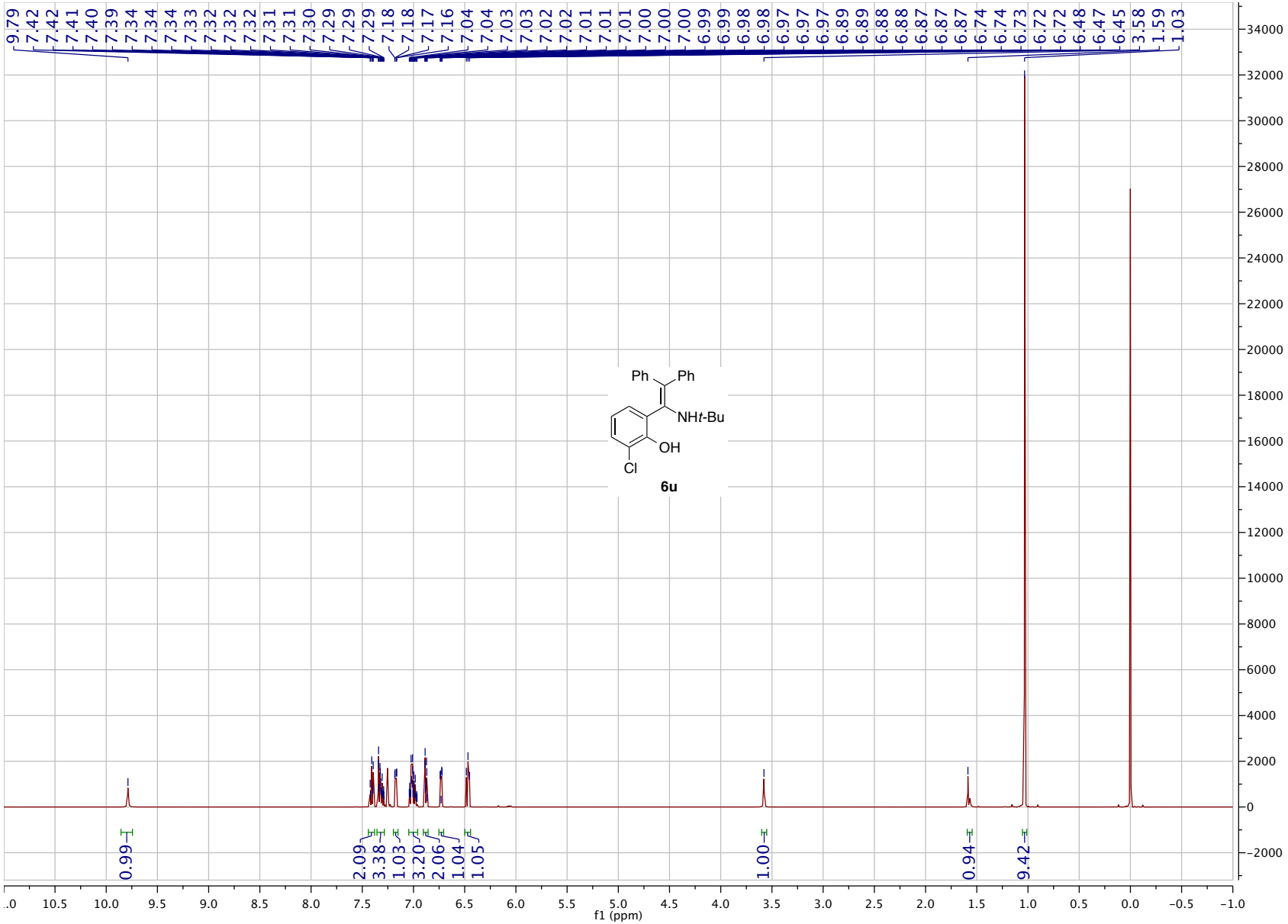
96.94
64.29
51.17
30.46
NHt-Bu
Ph
Ph
O
Cl
4u
f1 (ppm)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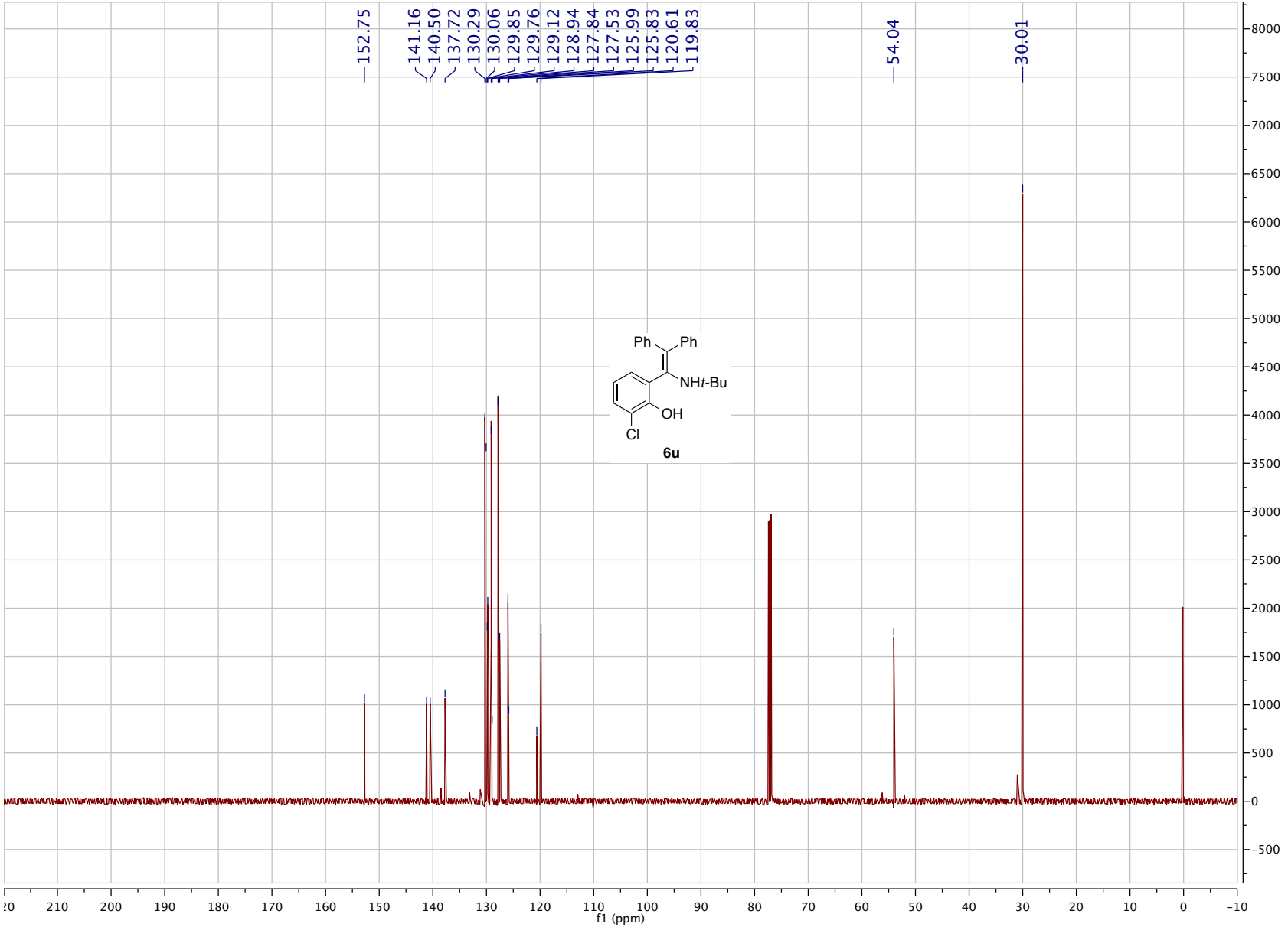
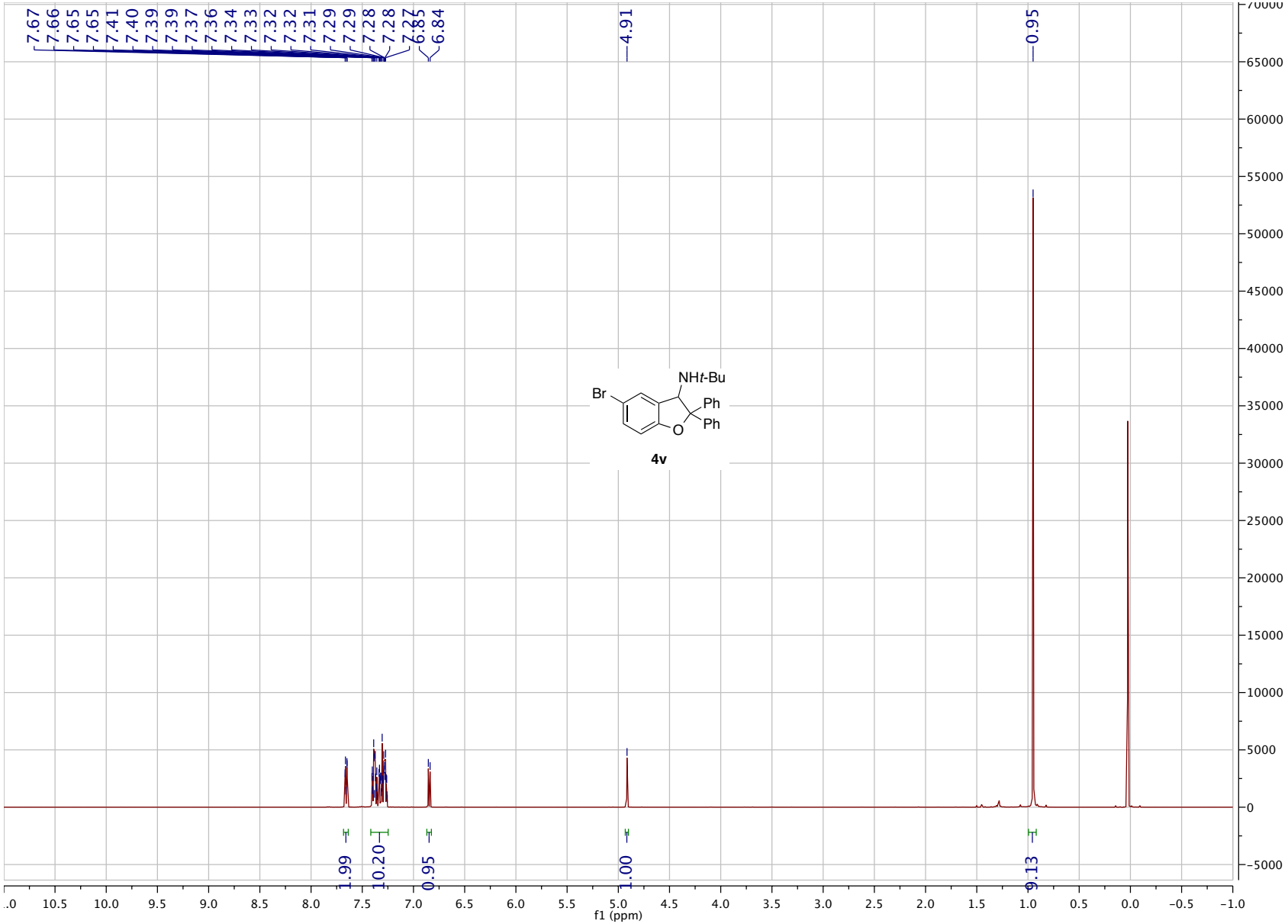
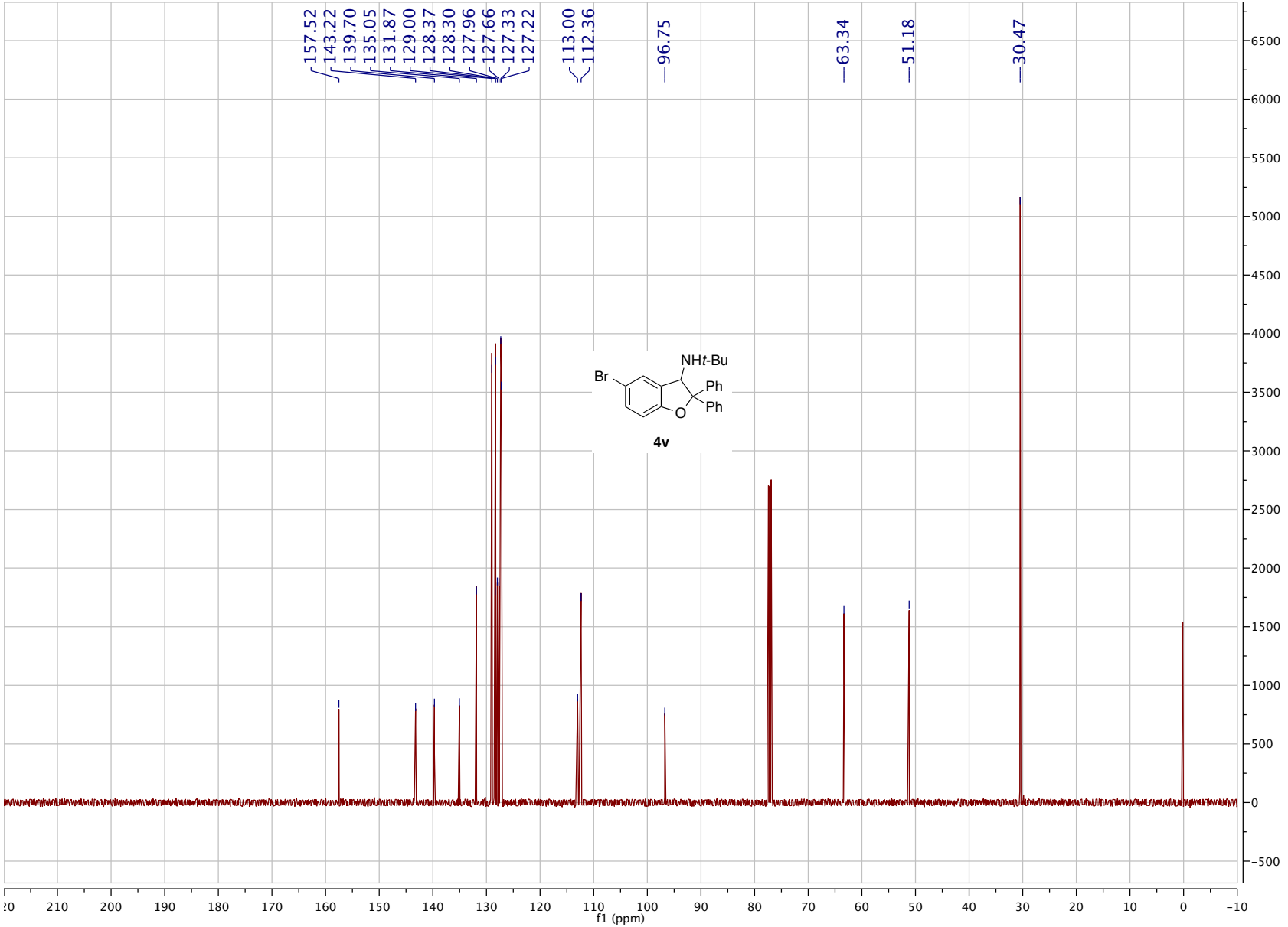
7.42
7.42
7.41
7.40
7.39
7.34
7.34
7.34
7.33
7.32
7.32
7.32
7.31
7.31
7.30
7.29
7.29
7.29
7.18
7.18
7.17
7.16
7.04
7.04
7.03
7.03
7.02
7.02
7.01
7.01
7.01
7.00
7.00
7.00
6.99
6.99
6.98
6.98
6.97
6.97
6.97
6.89
6.89
6.88
6.88
6.87
6.87
6.87
6.74
6.74
6.73
6.72
6.72
6.48
6.47
6.45
3.58
1.59
1.03
Ph
Ph
NHt-Bu
OH
Cl
6u
0.99
2.09
3.38
1.03
3.20
2.06
1.04
1.05
1.00
0.94
9.42
f1 (ppm)


152.75
141.16
140.50
137.72
130.29
130.06
129.85
129.76
129.12
128.94
127.84
127.53
125.99
125.83
120.61
119.83
54.04
30.01
Ph
Ph
NHt-Bu
OH
Cl
6u
f1 (ppm)


7.67
7.66
7.65
7.65
7.41
7.40
7.39
7.39
7.37
7.36
7.34
7.33
7.32
7.32
7.31
7.29
7.29
7.28
7.28
7.27
6.85
6.84
4.91
0.95
NHt-Bu
Br
Ph
Ph
O
4v
1.99
10.20
0.95
1.00
9.13
f1 (ppm)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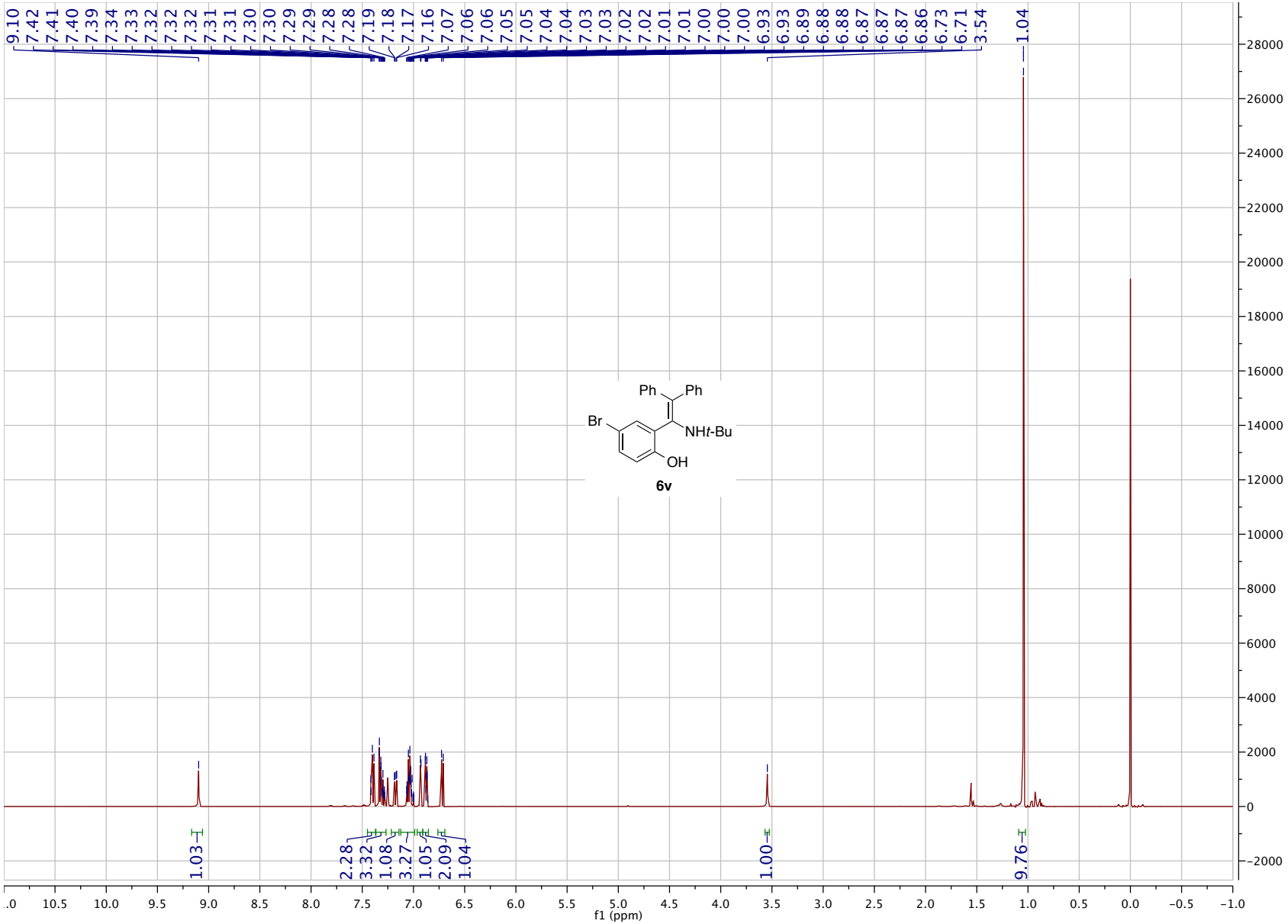
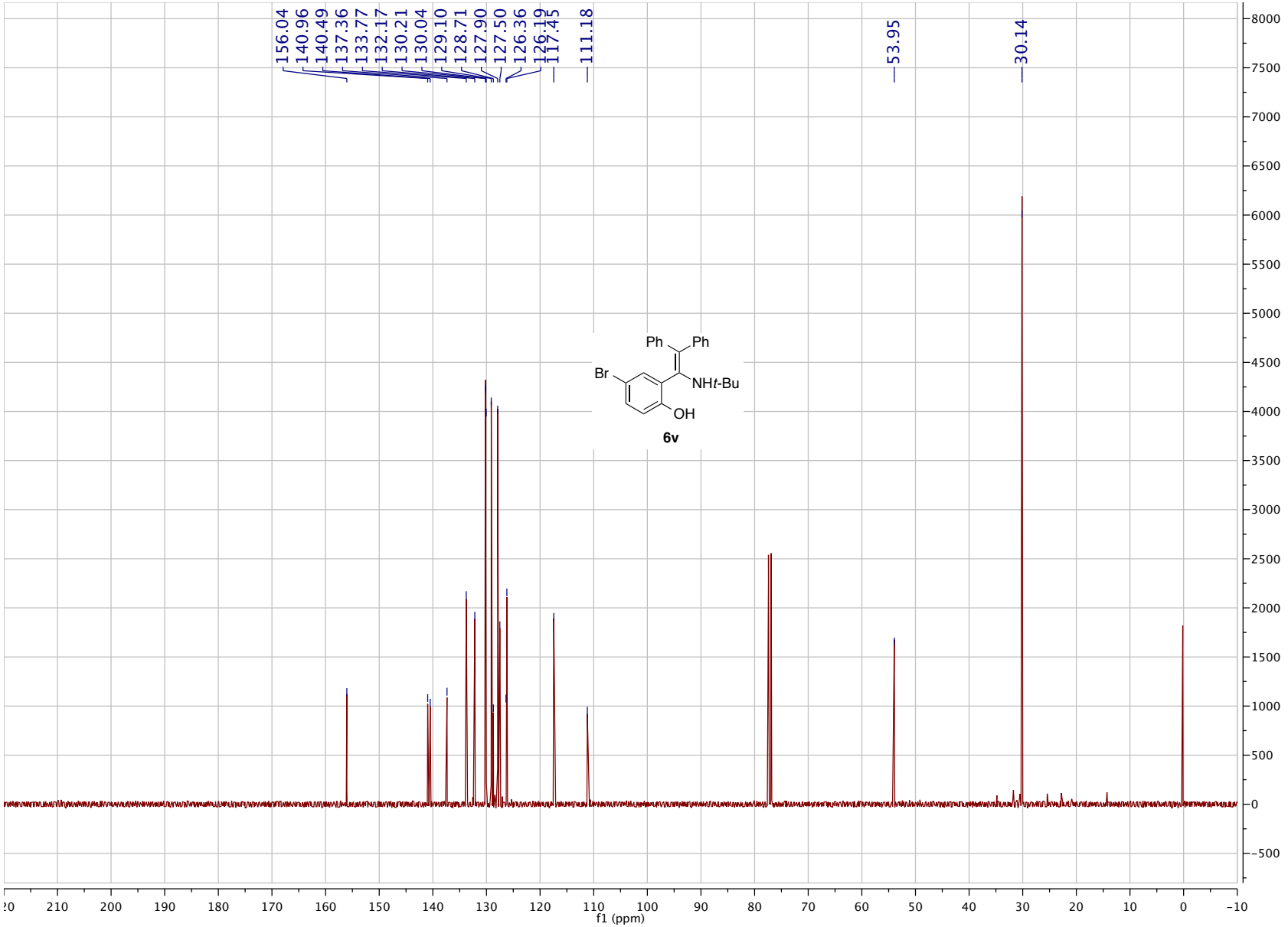
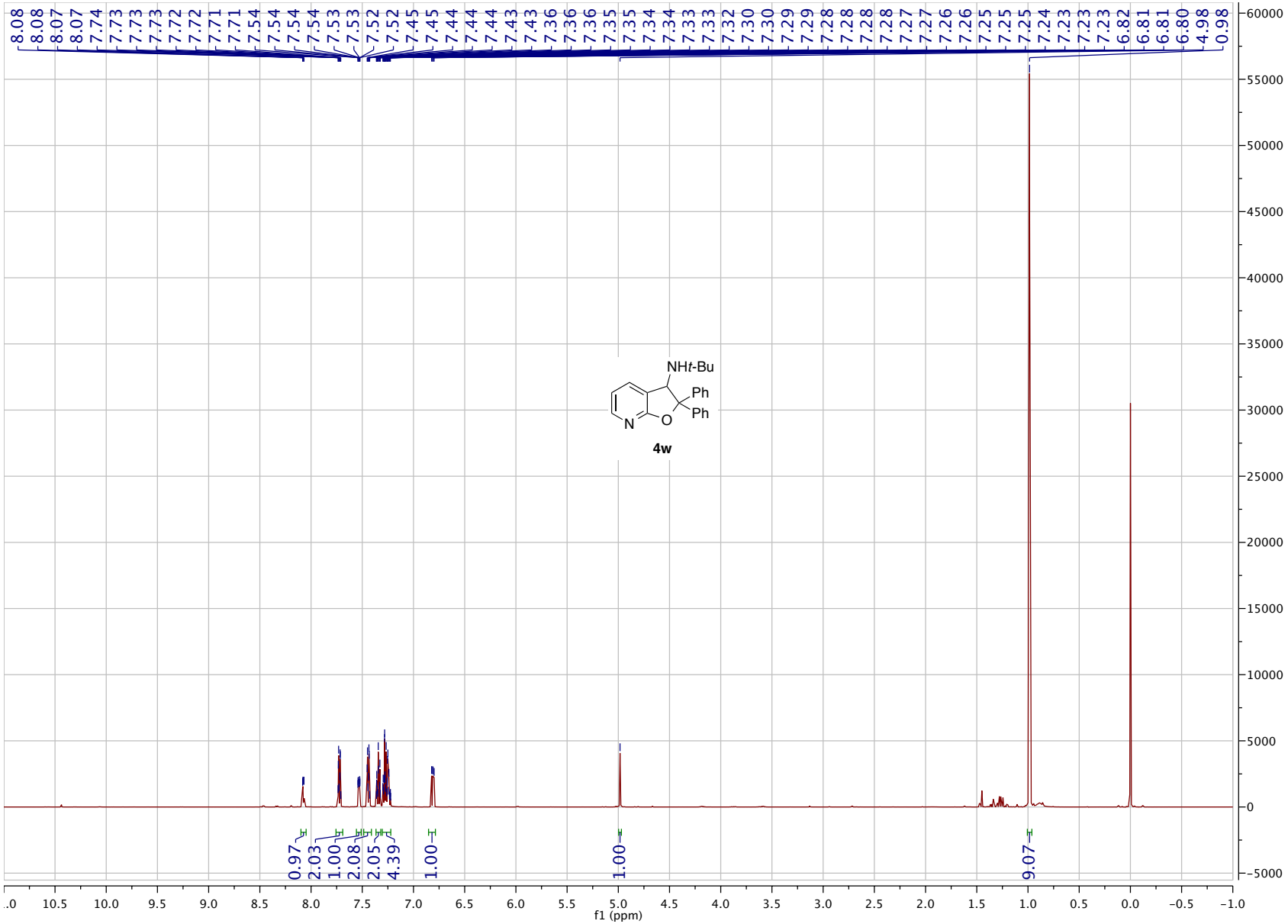
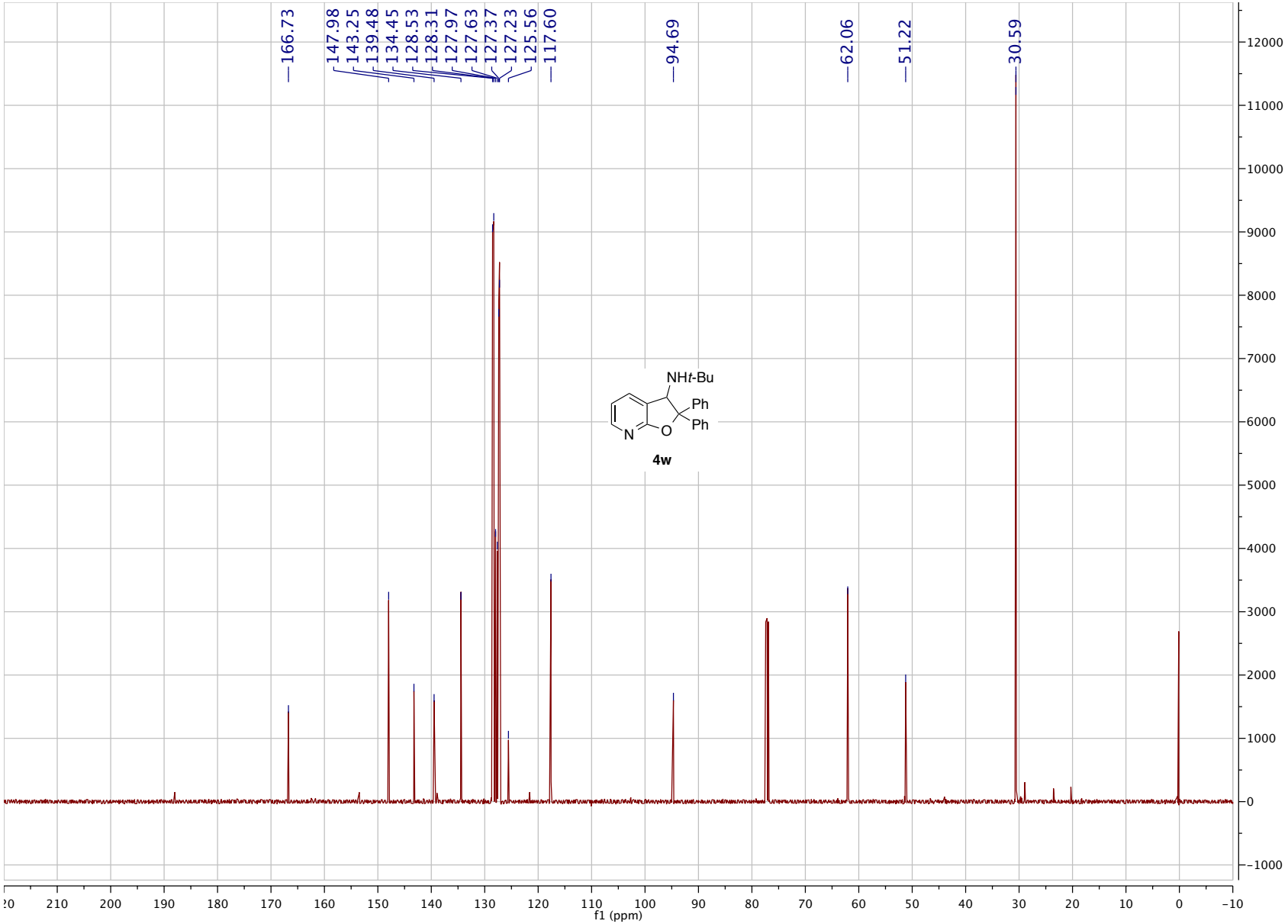
139.70
135.05
131.87
129.00
128.37
128.30
127.96
127.66
127.33
127.22
113.00
112.36
96.75
63.34
51.18
30.47
NHt-Bu
Br
Ph
Ph
O
4v
f1 (ppm)


9.10
7.42
7.41
7.40
7.39
7.34
7.33
7.32
7.32
7.32
7.31
7.31
7.30
7.30
7.29
7.29
7.28
7.28
7.19
7.18
7.17
7.16
7.07
7.06
7.06
7.05
7.05
7.04
7.04
7.03
7.03
7.02
7.02
7.01
7.01
7.00
7.00
7.00
6.93
6.93
6.89
6.88
6.88
6.87
6.87
6.87
6.86
6.73
6.71
3.54
1.04
Ph
Ph
Br
NHt-Bu
OH
6v
1.03
2.28
3.32
1.08
3.27
1.05
2.09
1.04
1.00
9.76
f1 (ppm)


156.04
140.96
140.49
137.36
133.77
132.17
130.21
130.04
129.10
128.71
127.90
127.50
126.36
126.19
117.45
111.18
53.95
30.14
Ph
Ph
Br
NHt-Bu
OH
6v
f1 (ppm)


8.08 8.08 8.07 8.07 7.74 7.73 7.73 7.73 7.72 7.72 7.71 7.71 7.54 7.54 7.54 7.54 7.53 7.53 7.52 7.52 7.45 7.45 7.44 7.44 7.44 7.44 7.43 7.43 7.36 7.36 7.36 7.35 7.35 7.34 7.34 7.33 7.33 7.32 7.30 7.30 7.29 7.29 7.28 7.28 7.28 7.28 7.27 7.27 7.26 7.26 7.25 7.25 7.25 7.24 7.23 7.23 6.82 6.81 6.81 6.80 4.98 0.98
NHt-Bu
Ph
Ph
N
O
4w
0.97 2.03 1.00 2.08 2.05 4.39 1.00 1.00 9.07
f1 (ppm)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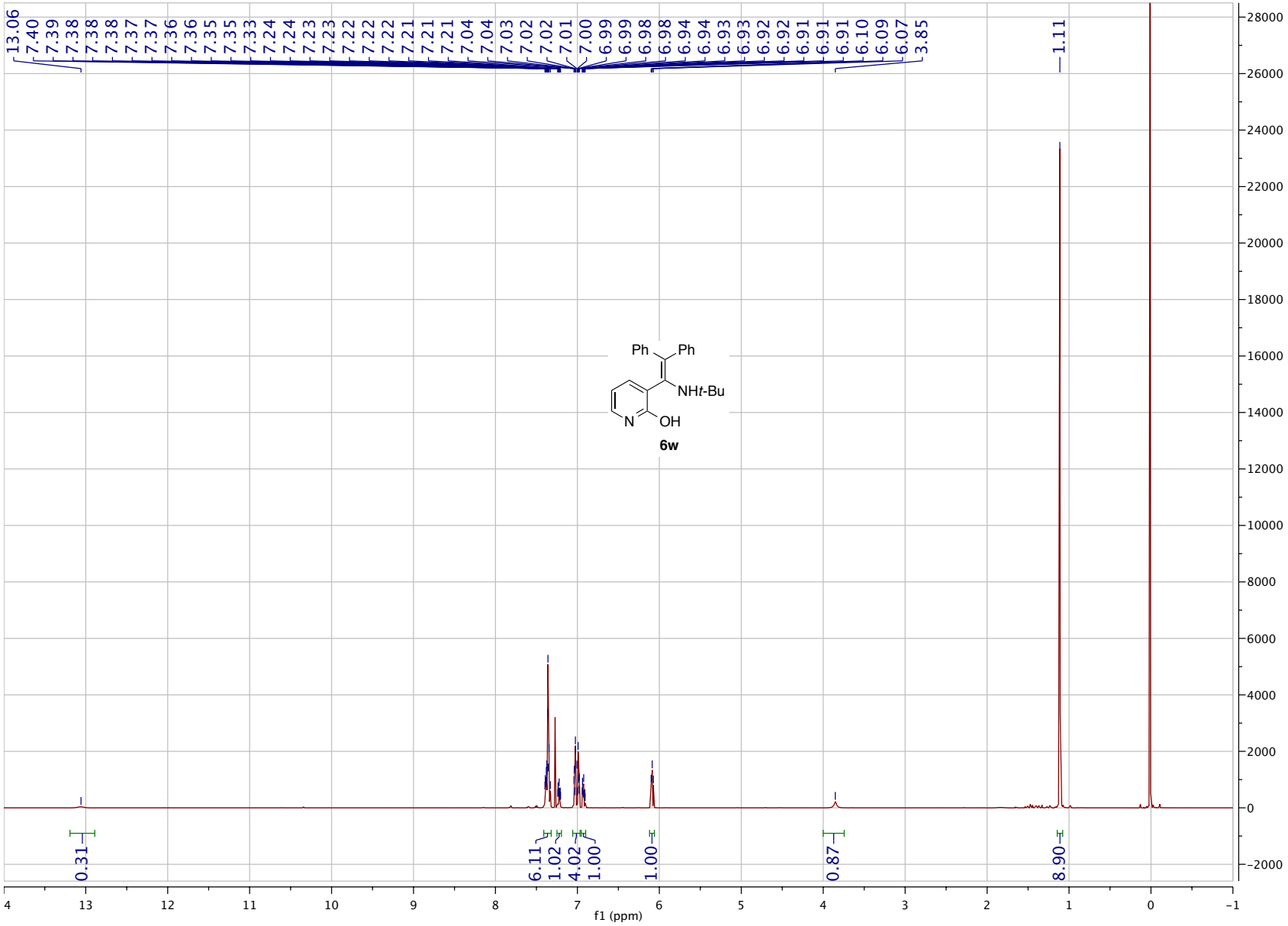
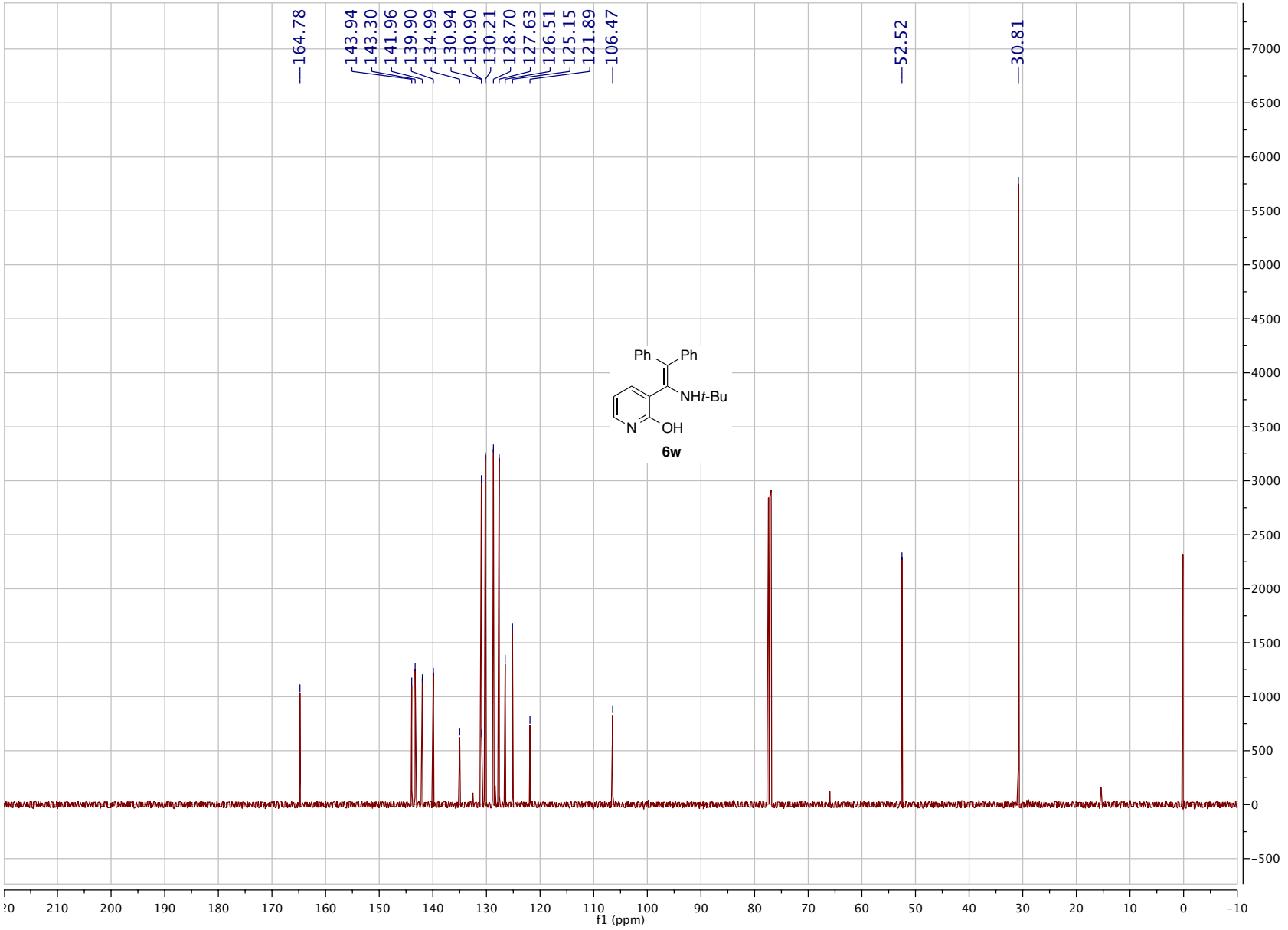
147.98
143.25
139.48
134.45
128.53
128.31
127.97
127.63
127.37
127.23
125.56
117.60
94.69
62.06
51.22
30.59
NHt-Bu
Ph
Ph
N
O
4w
f1 (ppm)


13.06
7.40
7.39
7.38
7.38
7.38
7.37
7.37
7.36
7.36
7.35
7.35
7.33
7.24
7.24
7.23
7.23
7.22
7.22
7.22
7.21
7.21
7.21
7.04
7.04
7.03
7.02
7.02
7.01
7.00
6.99
6.99
6.98
6.98
6.94
6.94
6.93
6.93
6.92
6.92
6.91
6.91
6.91
6.10
6.09
6.07
3.85
1.11
Ph
Ph
NHt-Bu
N
OH
6w
0.31
6.11
1.02
4.02
1.00
1.00
0.87
8.90
f1 (ppm)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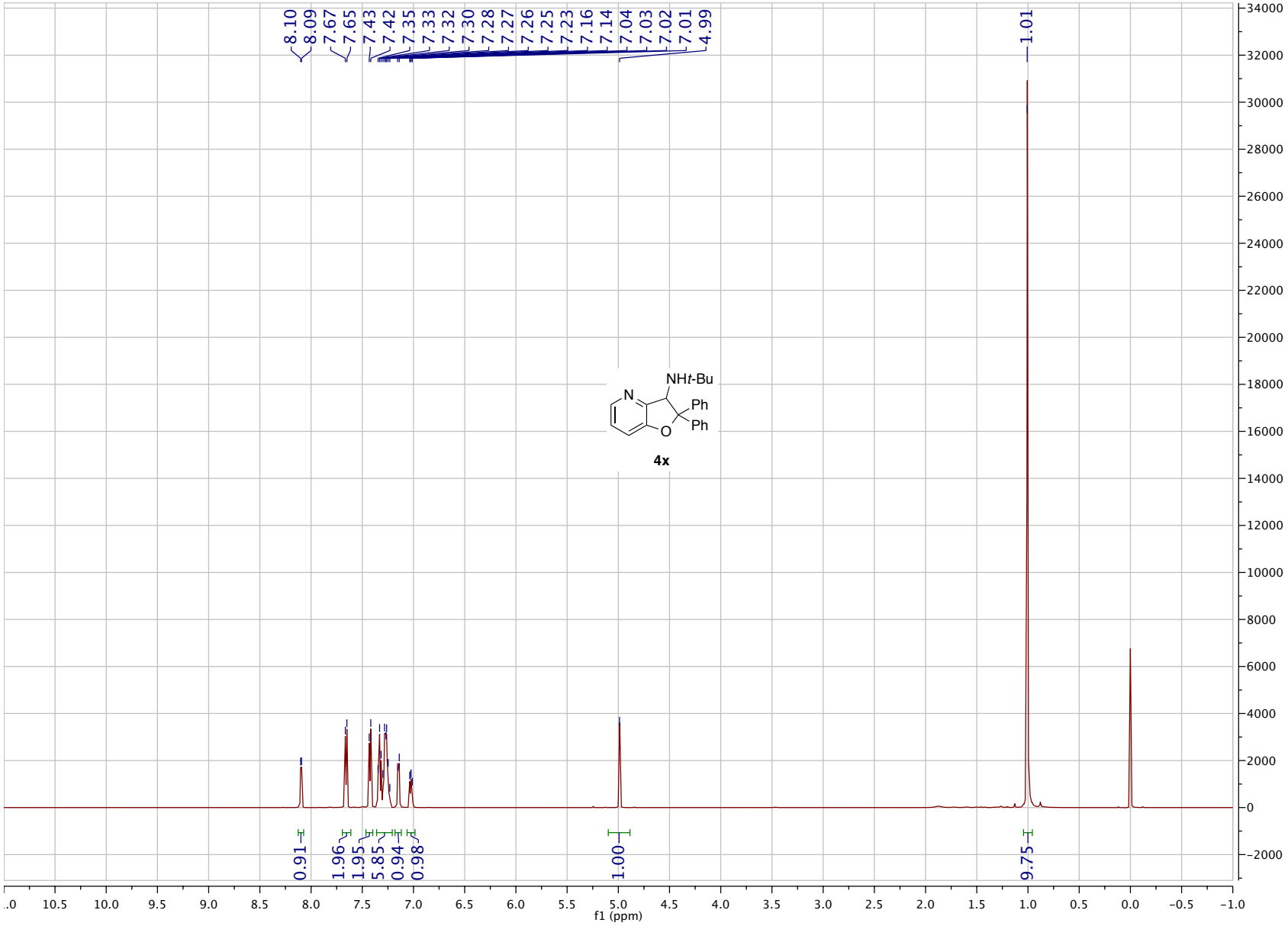
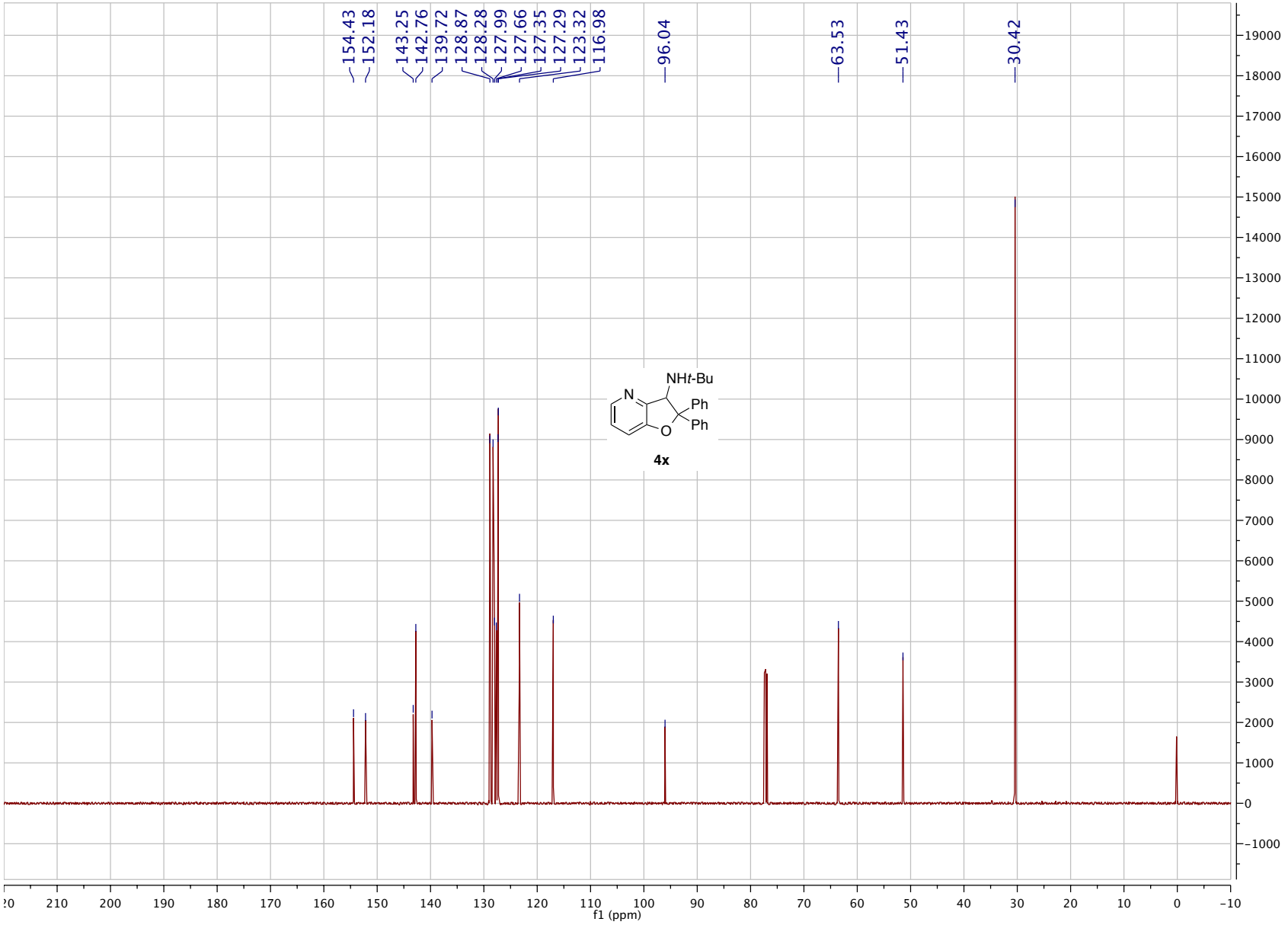
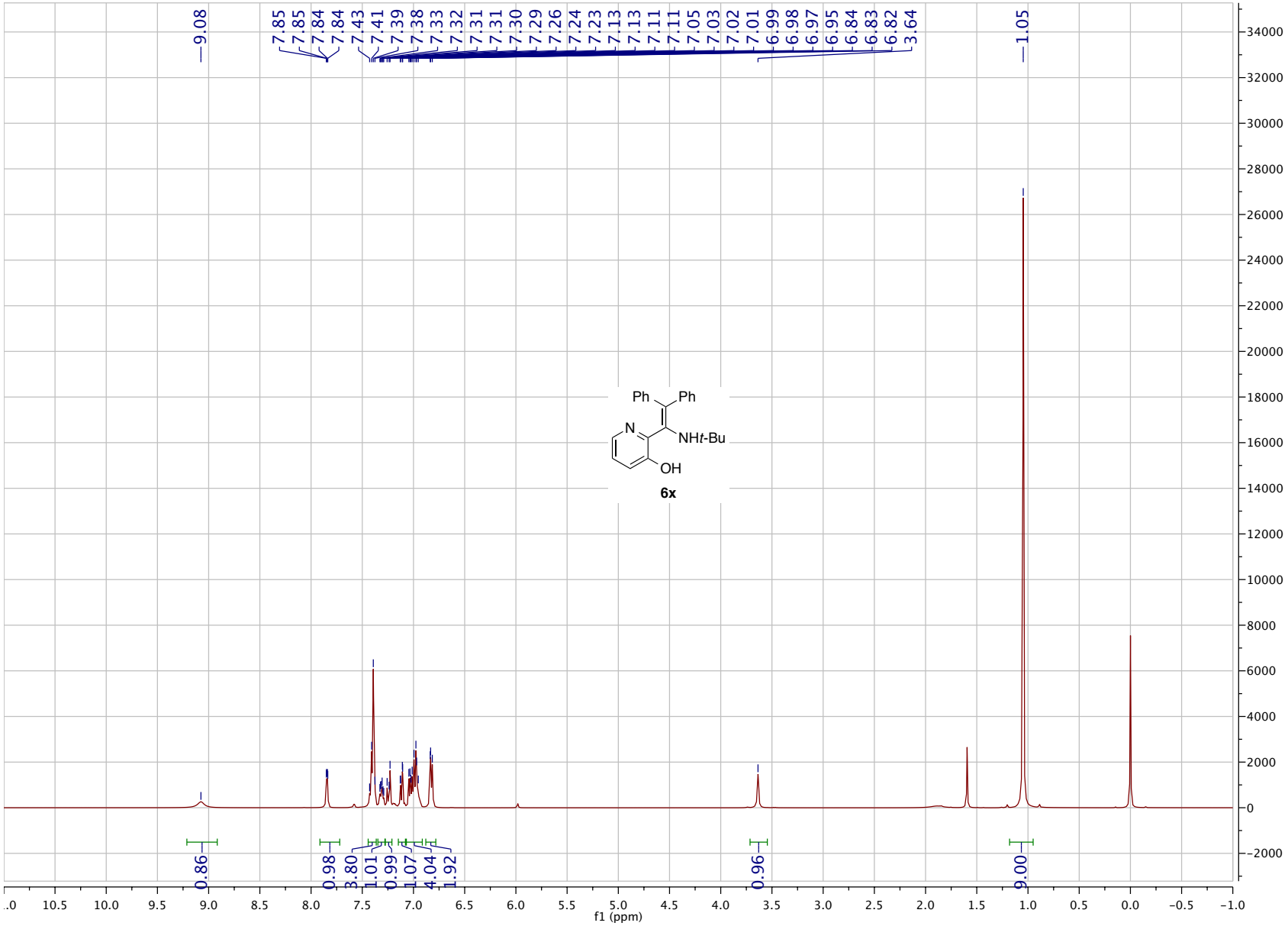
143.30
141.96
139.90
134.99
130.94
130.90
130.21
128.70
127.63
126.51
125.15
121.89
106.47
52.52
30.81
Ph
Ph
NHt-Bu
N
OH
6w
f1 (ppm)


8.10
8.09
7.67
7.65
7.43
7.42
7.35
7.33
7.32
7.30
7.28
7.27
7.26
7.25
7.23
7.16
7.14
7.04
7.03
7.02
7.01
4.99
1.01
NHt-Bu
N
Ph
Ph
O
4x
0.91
1.96
1.95
5.85
0.94
0.98
1.00
9.75
f1 (ppm)


154.43
152.18
143.25
142.76
139.72
128.87
128.28
127.99
127.66
127.35
127.29
123.32
116.98
96.04
63.53
51.43
30.42
NHt-Bu
N
Ph
Ph
O
4x
f1 (ppm)


9.08
7.85
7.85
7.84
7.84
7.43
7.41
7.39
7.38
7.33
7.32
7.31
7.31
7.30
7.29
7.26
7.24
7.23
7.13
7.13
7.11
7.11
7.05
7.03
7.02
7.01
6.99
6.98
6.97
6.95
6.84
6.83
6.82
3.64
1.05
Ph
Ph
N
NHt-Bu
OH
6x
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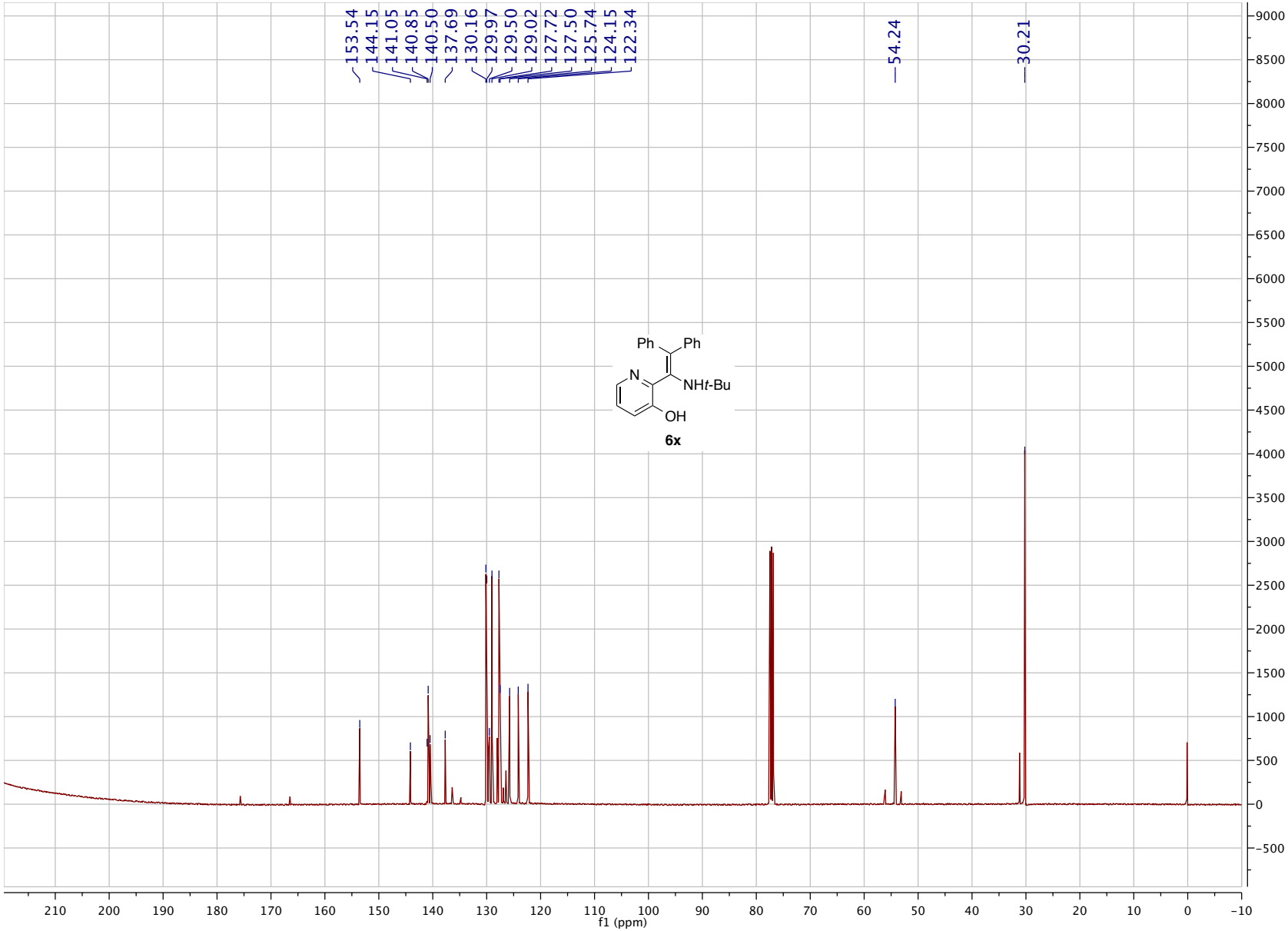
0.98
3.80
1.01
0.99
1.07
4.04
1.92
0.96
9.00
f1 (ppm)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144.15
141.05
140.85
140.50
137.69
130.16
129.97
129.50
129.02
127.72
127.50
125.74
124.15
122.34
54.24
30.21
Ph
Ph
N
NHt-Bu
OH
6x
f1 (ppm)